EFFICIENT SYNTHESIS OF HOMOALLYLIC ALCOHOLS/AMINES FROM ALLYLTRIBUTYLSTANNANE AND CARBONYL COMPOUNDS/IMINES USING IODINE AS CATALYST UNDER ACETIC ACID–WATER MEDIUM

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GRAPHICAL ABSTRACT

Abstract
This paper describes a general method for the synthesis of homoallylic alcohols and amines by nucleophilic addition reaction of allylttributylstannane to carbonyl compounds and aldmines where iodine acts as a catalyst in H2O/acid (1:1) medium. Only 10 mol% of I2 is required for various organic transformations. By using this process, various homoallylic alcohols and amines are produced in good to excellent yields.

Keywords
Aldimines; allylation; carbonyl compounds; iodine

INTRODUCTION

Allylation of carbonyl compounds and aldmines to produce homoallylic alcohols and amines respectively has been widely investigated and established as a powerful tool to synthesize valuable intermediates such as β-hydroxy carbonyl compounds, β- and γ-amino acids, spiro cyclic compounds, and γ-lactams. They have attracted significant attention because of their versatile uses as important building blocks for natural product synthesis.[1] Hence, numerous reagents and methods have been developed to accomplish this transformation.[2] A number of methods have been developed for allylation reaction involving various metals[3] such as zinc, tin, indium, bismuth, manganese, magnesium, antimony, scandium,
cobalt, cerium, and silver. Killinger and coworkers\[4\] reported the allylation of aldehydes and ketones with allyl bromide mediated by activated zinc dust in 95% ethanol at 78 °C. Araki et al. reported the allylation of carbonyl compounds using indium metal in an anhydrous organic solvent.\[5\] Later, Li and Chan successfully carried out the indium-mediated, Barbier-type allylation in aqueous medium.\[6\] Nokami et al.\[7\] observed that water had an accelerating effect on the allylation of carbonyl compounds with diallyltin dibromide in ether. Chen\[8\] reported Sb-mediated allylation in alcohol-water mixture. Barczak and Jarvo.\[9\] reported silver bromide–catalyzed allylation of aldehydes and ketones using unactivated manganese powder. In the same year Wu and Sun\[10\] reported the facile allylation of N-Boc and N-Cbz imines by using allyltrichlorosilane promoted by dimethylformamide (DMF) and acetonitrile at 0 °C. Zha\[11\] and coworkers used tin nanoparticles for the allylation of aldehydes and ketones in aqueous medium. Slaton et al.\[12\] reported tin-mediated Barbier-type allylation in ionic liquids. In the same year, Li and coworkers\[13\] reported a mild and efficient allylation reaction mediated by magnesium powder under solvent-free conditions. Tan et al.\[14\] reported a method for the allylation reaction of the carbonyl compounds using 2 molar equiv. of Cu powder to accomplish the product formation. Bellucci et al.\[15\] developed a method for the allylation of imines promoted by lanthanide triflates. One year later, Wang\[16\] and coworkers reported that imines can be allylated by magnesium- or zinc-mediated Barbier-type procedure to produce homoallylamines in excellent yields. Phukan\[17\] reported that only 10 mol% iodine catalyzes efficiently the three-component reaction of aldehydes, benzyl carbamate, and allyltrichlorosilane to afford the corresponding protected homoallylamine in good to excellent yield. Yadav et al.\[18\] reported the allylation and alkynylation of cyclic allylic acetates with allyltrichlorosilane and alkynyl silanes in the presence of molecular iodine in dichloromethane at rt.

Although a number of allylating reagents are used for the allylation reaction, allyltributylstannane is one of the most common reagents used for the synthesis of homoallylic alcohols and amines. Generally, strong Lewis acids such as TiCl4 and BF3 · OEt2 and other metal complexes such as PdCl2(PPh3)2, PtCl2(PPh3)2, bis-π-allylpalladium complex, lanthanide salts, and LiClO4 are used for the preparation of homoallylic amines.\[19\] However, in most of allylation reactions of carbonyl compound, either poor yield\[12,14\] or prolonged reaction time\[9\] is needed. Also, metal triflates are highly expansive.\[15\] Therefore, the development of novel, common, cheap, high-yielding, and useful catalytic methods is of interest. Iodine has emerged as a very effective catalyst for various organic transformations.\[20\] In continuation of our work on various allylation reaction,\[21\] we report herein a method for the synthesis of homoallylic alcohols and amines by nucleophilic addition reaction

$$X \overset{\text{SnBu}_3}{\text{R}^1 \text{R}^2} + \text{Acetic acid: H}_2\text{O (1:1)} \rightarrow \text{I}_2 / 35^\circ\text{C} \rightarrow \text{XH} \text{R}^1 \text{R}^2 \text{R}^3$$

\(R^1 = \text{Ar}\)
\(X = \text{O, NR}_2\)
\(R^2 = \text{H, CH}_3\)

Scheme 1 Allylation of carbonyl compounds and aldimines with allyltributylstannane.
of allyltributylstannane to carbonyl compounds and aldimines using I₂ as catalyst in the presence of H₂O/acetic acid (1:1) (Scheme 1).

RESULTS AND DISCUSSION

In a typical reaction, aldehydes (1 mmol), allyltributylstannane (1 mmol), and I₂ (5 mol%) were added successively in H₂O/acetic acid (1:1; 1 mL) at room temperature. The reaction was monitored by thin-layer chromatography (TLC). The results are presented in Table 1. Initial experiments without acetic acid failed to produce any product at room temperature (Table 1, entry 1). Another experiment without both H₂O and the catalyst also produced a negative result (Table 1, entries 2 and 3 respectively). When the reaction was examined using iodine as catalyst (5 mol%), in a mixed solvent system H₂O–acetic acid at the ratio of 1:1, it produced 58% of the product formation after 1 h at room temperature (Table 1, entry 4). The reaction, with increasing the concentration of the catalyst as well as allyltributylstannane, gradually increased the yield of the reaction up to 95%. However, the reaction showed positive results when we examined the reaction by using some Lewis acids, e.g., CuI and FeCl₃, as catalyst, where the corresponding homoallyl alcohols produced moderate to poor yield, respectively (Table 1, entries 14 and 15). The reaction was also examined using different solvents such as tetrahydrofuran (THF), CH₃CN, dimethylformamide (DMF), dimethylsulfoxide (DMSO), and CH₂Cl₂ (Table 1, entries 9–13). In every case, the reaction was found to be inactive for product formation. Finally, the use of 10 mol% of I₂, 1.2 equiv of allyltributylstannane, and 1 equiv of benzaldehyde in H₂O/acetic acid (1:1; 1 mL), was found to be optimum to realize the best yield of the product.

After establishing the optimized reaction conditions, a variety of aromatic and aliphatic aldehydes were investigated under the same reaction conditions. The results are summarized in Table 2. In general, the procedure works well for a variety of

| Entry | PhCHO (mmol) | Solvent (mL) | Acetic acid (mL) | Allyl tributylstannane (mmol) | Catalyst (mol%) | Time (h) | Yield (%) |
|-------|--------------|--------------|-----------------|-----------------------------|----------------|---------|----------|
| 1     | 1            | H₂O (1)      | —               | 1                           | I₂ (5)         | 24      | 0        |
| 2     | 1            | —            | 1               | I₂ (5)                      | 24             | 0       |
| 3     | 1            | H₂O (1)      | 1               | I₂ (5)                      | 24             | 0       |
| 4     | 1            | H₂O (1)      | 1               | I₂ (5)                      | 1              | 58      |
| 5     | 1            | H₂O (1)      | 1               | I₂ (5)                      | 1              | 82      |
| 6     | 1            | H₂O (1)      | 1               | I₂ (5)                      | 1              | 90      |
| 7     | 1            | H₂O (1)      | 1               | I₂ (10)                     | 1              | 90      |
| **8** | **1**        | **H₂O (1)**  | **1**           | **I₂ (10)**                 | **1**          | **95**  |
| 9     | 1            | THF (1)      | —               | I₂ (10)                     | 24             | 0       |
| 10    | 1            | CH₃CN (1)    | —               | I₂ (10)                     | 24             | 0       |
| 11    | 1            | DMF (1)      | —               | I₂ (10)                     | 24             | 0       |
| 12    | 1            | DMSO (1)     | —               | I₂ (10)                     | 24             | 0       |
| 13    | 1            | CH₂Cl₂ (1)   | —               | I₂ (10)                     | 24             | 0       |
| 14    | 1            | H₂O (1)      | 1               | CuI (10)                    | 1              | 40      |
| 15    | 1            | H₂O (1)      | 1               | FeCl₃ (10)                  | 1              | 10      |

*Isolated yield after chromatographic purification.*
Table 2. Acetic acid/H$_2$O-mediated and I$_2$-catalyzed allylation of aldehydes

| Entry | Aldehydes/ketones | Product\(^b\) | Time (h) | Yield (%)\(^c\) |
|-------|------------------|--------------|----------|----------------|
| 1     | \(\text{PhCHO}\) | \(\text{3a}\) | 1.0      | 95             |
| 2     | \(\text{Cl-PhCHO}\) | \(\text{3b}\) | 1.0      | 83             |
| 3     | \(\text{MeO-PhCHO}\) | \(\text{3c}\) | 1.0      | 88             |
| 4     | \(\text{OMe-PhCHO}\) | \(\text{3d}\) | 1.0      | 92             |
| 5     | \(\text{NO$_2$-PhCHO}\) | \(\text{3e}\) | 1.0      | 76             |
| 6     | \(\text{NO$_2$-PhCHO}\) | \(\text{3f}\) | 1.0      | 87             |
| 7     | \(\text{H$_3$C-PhCHO}\) | \(\text{3g}\) | 1.0      | 85             |
| 8     | \(\text{Cl-PhCHO}\) | \(\text{3h}\) | 1.0      | 93             |
| 9     | \(\text{Cl-PhCHO}\) | \(\text{3i}\) | 1.0      | 88             |

(Continued)
Table 2. Continued

| Entry | Aldehydes/ketones      | Product<sup>b</sup> | Time (h) | Yield (%)<sup>c</sup> |
|-------|------------------------|---------------------|----------|-----------------------|
|       | Br-CHO                 | 3j                  | 1.0      | 83                    |
|       | CH-CHO                 | 3k                  | 2.0      | 79                    |
|       | Br-CHO                 | 3l                  | 2.0      | 82                    |
|       | O-CHO                  | 3m                  | 2.0      | 76                    |
|       | F-CHO                  | 3n                  | 2.2      | 93                    |
|       | O-CHO                  | 3o                  | 2.0      | 83                    |
|       | O-CHO                  | 3p                  | 2.5      | 85                    |
|       | O-CHO                  | 3q                  | 2.2      | 83                    |
|       | O-CHO                  | 3r                  | 2.5      | 82                    |

<sup>a</sup>Reaction condition: Aldehydes/ketones, 1 mmol; allyltributylstannane, 1.2 mmol; \( \text{H}_2\text{O}/\text{AcOH} = 1:1; \) \( I_2, 10 \text{ mol} \%. \)

<sup>b</sup>All products were characterized by IR, \( ^1\text{H} \) NMR, and \( ^{13}\text{C} \) NMR.

<sup>c</sup>Isolated yield after chromatographic purification.
Table 3. I$_2$-catalyzed three-component synthesis of homoallylic amines

$$\begin{align*}
\text{R}^1\text{COH} + \text{Ph}^\text{NH}_2 + \text{SnBu}_3^+ & \xrightarrow{\text{Acetic acid: H}_2\text{O (1:1)}} \text{I}_2 / \text{rt} \quad \text{R}^2\text{NH}^\text{Ph} \\
4 & \quad 5 & \quad 2 & \quad 6a-j
\end{align*}$$

| Entry | Reactant | Product$^a$ | Time (h) | Yield (%)$^c$ |
|-------|----------|-------------|----------|---------------|
| 1     | \(\text{O}\) | 6a          | 2.0      | 83            |
| 2     | \(\text{O}_2\) | 6b          | 2.5      | 92            |
| 3     | \(\text{Me}\) | 6c          | 2.0      | 73            |
| 4     | \(\text{Cl}\) | 6d          | 2.0      | 84            |
| 5     | \(\text{Ph-CHO}\) | 6e          | 2.0      | 77            |
| 6     | \(\text{Cl}\) | 6f          | 2.5      | 88            |
| 7     | \(\text{NO}_2\) | 6g          | 2.5      | 89            |
| 8     | \(\text{Cl}\) | 6h          | 2.0      | 82            |

(Continued)
aliphatic as well as aromatic aldehydes to produce the corresponding homoallylalcohols in excellent yield. Substituted aromatics bearing the groups –Cl, -OMe, -Br, -NO2, and -CH3 reacted very successfully and no matter the substituent position in the aromatic ring produced good yields.

The success of the homoallylation of the aldehydes prompted us to extend the procedure to ketones (Table 2, entries 13–18) and aldimines (Table 3), which were produced in situ of the reaction between aldehydes and amines. Initial experiment with acetophenone produced 76% yield after 2 h (Table 2, entry 13) of the reaction time. The reaction was further extended to a variety of ketones in which cases aromatic, cyclic, and aliphatic ketones all produced excellent yields with slightly longer reaction times.

### CONCLUSION

In summary, we have developed a method for allylation reaction of aldehydes, ketones, and imines with allyltrIBUTYlstannane in acetic acid–water medium. The advantages of these methods include the use of a nontoxic catalyst, easy handling, and generation of homoallyl alcohol and amine in one step. Further investigation regarding the asymmetric version of this reaction in the same solvent system is currently in progress in our laboratory.

### EXPERIMENTAL

All the chemicals used were of analytical grade. 1H and 13C NMR spectra were obtained in CDCl3 at 300 and 75 MHz, respectively, using a Bruker Ultrasound 300-MHz spectrometer (with tetramethylsilane, TMS, as internal reference). Mass spectra were recorded on a Perkin-Elmer Clarus 600 C mass spectrometer. Infrared (IR) spectra were recorded on a Perkin-Elmer Spectrum RX IFT-IR spectrometer.
Synthesis of 1-Phenyl-but-3-en-1-ol

A mixture of benzaldehyde (106 mg, 1 mmol), allyltributylstannane (397 mg, 1.2 mmol), and I₂ (25.3 mg, 0.1 mmol) was added in H₂O/AcOH (1:1, 1 mL), at room temperature. After stirring the reaction for the appropriate time (checked by thin-layer chromatography, TLC), the solution was added to water and the reaction mixture was extracted with diethylether (3 × 10 mL). The combined organic extract was dried over sodium sulfate, filtered, and concentrated. The crude product was purified by flash column chromatography over silica gel (230–400 mesh) using ethylacetate–petroleum ether (5:95) as eluent.

1-Phenyl-but-3-en-1-ol (3a)

IR (v/cm⁻¹): 3372, 3073, 2907, 1641, 1493, 1445, 1043; ¹H NMR (300 MHz, CDCl₃): δ 7.43–7.23 (m, 5H), 5.91–5.72 (m, 1H), 5.24–5.11 (m, 2H), 4.80–4.70 (m, 1H), 2.60–2.45 (m, 2H), 2.00 (s, 1H); ¹³C NMR (75 MHz, CDCl₃): δ 143.8, 134.4, 128.4, 127.5, 125.8, 118.5, 73.3, 43.8; ESIMS (m/z) 149 [M+H]⁺.

Benzyl-(1-phenyl-but-3-enyl)-amine (6a)

IR (v/cm⁻¹): 3460, 3058, 2921, 1489; ¹H NMR (300 MHz, CDCl₃): δ 7.97–7.01 (m, 9H), 5.80–5.60 (m, 1H), 5.18–4.92 (m, 2H), 4.22–4.10 (m, 1H), 3.46 (d, J = 12 Hz, 1H), 3.40 (d, J = 12 Hz, 1H), 2.60 (s, 1H, NH), 2.40–2.10 (m, 2H); ESIMS (m/z) 238 [M+H]⁺.

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