

## Research Article

# Regioselective and Chemoselective Reduction of $\alpha,\beta$ -Unsaturated Carbonyl Compounds by $\text{NaBH}_4/\text{Ba}(\text{OAc})_2$ as a Reducing System

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Received 24 February 2013; Revised 12 April 2013; Accepted 12 April 2013

Academic Editor: Robert Salomon

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$\alpha,\beta$ -unsaturated aldehydes and ketones are regioselectively reduced to the corresponding allylic alcohols with  $\text{NaBH}_4/\text{Ba}(\text{OAc})_2$  system in  $\text{CH}_3\text{CN}$ . This system is also efficient for the chemoselective reduction of enals in the presence of enones at room temperature.

## 1. Introduction

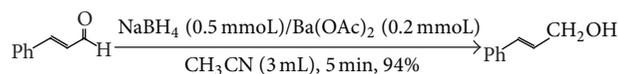
Reduction of  $\alpha,\beta$ -unsaturated carbonyl compounds widely has been carried out by different reducing agents. This reaction is highly solvent dependent and generally does not result in a useful regioselectivity [1–3]. It can follow two pathways: addition to carbonyl group (1,2-reduction) to give allylic alcohols or addition to the conjugated double bond (1,4-addition) to give saturated carbonyl compounds. To control the reducing potential and selectivity of metal hydrides specially  $\text{NaBH}_4$  (common reducing agent) for the 1,2-reduction of conjugated carbonyl compounds, numerous hydroborate agents have been developed in the following ways: (a) by the replacement of hydride(s) with bulky substituents or electron-withdrawing/releasing groups in order to discriminate between the structural and electronic environments of carbonyl groups [4–7]; (b) combination with Lewis acids [8–10] such as Luche reduction [11, 12] and mixed solvent systems [1, 2]; (c) use of transition metal hydroborates and their new modifications [13], (d) use of phosphonium tetrahydroborates [14, 15], and finally (e) immobilization on an anion exchange resin [16]. In this context and continuation of our studies for the reduction of functional groups in organic synthesis [17–20], we decided to apply

$\text{NaBH}_4/\text{Ba}(\text{OAc})_2$  as new a reducing system for reduction of  $\alpha,\beta$ -unsaturated carbonyl compounds. Now we wish to report an efficient method for the regioselective reduction of  $\alpha,\beta$ -unsaturated aldehydes and ketones by  $\text{NaBH}_4/\text{Ba}(\text{OAc})_2$  as a new reducing system.

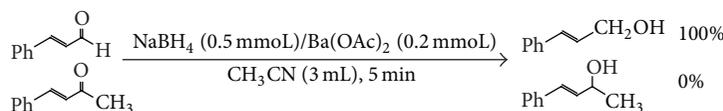
## 2. Experimental

**2.1. General.** All substrates and reagents were purchased from commercial sources with the best quality and used without further purification. IR and  $^1\text{H}$  NMR spectra were recorded on PerkinElmer FT-IR RXI and 300 MHz Bruker spectrometers, respectively. The products were characterized by their  $^1\text{H}$  NMR or IR spectra and comparison with authentic samples (melting or boiling points). Organic layers were dried over anhydrous sodium sulfate. All yields referred to isolated pure products. TLC was applied for the purity determination of substrates, products, and reaction monitoring over silica gel 60 F<sub>254</sub> aluminum sheet.

**2.2. A Typical Procedure for Regioselective 1,2-Reduction of Conjugated Carbonyl Compounds with  $\text{NaBH}_4/\text{Ba}(\text{OAc})_2$  as a New Reducing System.** In a round-bottomed flask (10 mL)



SCHEME 1



SCHEME 2

equipped with a magnetic stirrer and a condenser, a solution of benzylideneacetone (0.146 g, 1 mmol) and  $\text{Ba(OAc)}_2$  (0.05 g, 0.2 mmol) in  $\text{CH}_3\text{CN}$  (3 mL) was prepared, and  $\text{NaBH}_4$  (0.076 g, 2 mmol) was added. The resulting mixture was stirred under reflux conditions. TLC monitored the progress of the reaction (eluent; *n*-hexan/EtOAc: 9/1). After completion of the reaction within 15 min, distilled water (5 mL) was added to the reaction mixture and it was stirred for an additional 5 min. The mixture was extracted with  $\text{CH}_2\text{Cl}_2$  ( $3 \times 8$  mL) and dried over anhydrous sodium sulfate. Evaporation of the solvent afforded the pure 4-phenyl-3-buten-2-ol (0.141 g, 95% yield, Table 2, entry 3).

### 3. Results and Discussions

Due to the importance of synthetic precursors of allylic alcohols, the regioselective reduction of  $\alpha,\beta$ -unsaturated aldehydes and ketones seems to be a convenient and easy way to obtain these compounds. So, this achievement is synthetically very important. We first examined the reduction of cinnamaldehyde as a model compound. The reduction reaction took place with 0.5 molar amounts of  $\text{NaBH}_4$  in the presence of 0.2 molar amounts of  $\text{Ba(OAc)}_2$  in  $\text{CH}_3\text{CN}$  at room temperature. The reaction was completed within 5 min with a complete regioselectivity (Table 1, entry 2). The product, cinnamyl alcohol, was obtained in high yield (Table 2, entry 1) as shown in Scheme 1.

This procedure was also applied for the reduction of citral to geraniol (3,7-dimethyl-2,6-octadien-1-ol) which was obtained regioselectively in 93% yield. In the next attempt, we examined the reductions of conjugated enones with the  $\text{NaBH}_4/\text{Ba(OAc)}_2$  reducing system. The results showed that our procedure was also regioselective and efficient, but reduction reactions were performed by using 2 molar amounts of  $\text{NaBH}_4$  in the presence of 0.2 molar amounts of  $\text{Ba(OAc)}_2$  in  $\text{CH}_3\text{CN}$  under reflux conditions (Table 1, entry 9). Regioselective 1,2-reductions of benzalacetone, benzalacetophenone,  $\beta$ -ionone, 2-cyclohexenone, and 3-methyl-2-cyclopentenone were achieved successfully with high to excellent yields of the corresponding allylic alcohols (Table 2, entries 3–7).

The chemoselective reduction of one functional group without affecting the other one is a well-known strategy for preparing molecules with ever-increasing complexity in organic synthesis. Since the reduction of enals and enones with  $\text{NaBH}_4$  in the presence of  $\text{Ba(OAc)}_2$  is dependent on molar ratio of  $\text{NaBH}_4$  and temperature, therefore, we thought

that this system has a chemoselectivity towards reduction of enals over enones. The chemo- and regioselectivity of this procedure were demonstrated by a competitive reduction of cinnamaldehyde over benzalacetone as shown in Scheme 2.

The usefulness of this chemo- and regioselectivity of the reduction was further examined with the reduction of cinnamaldehyde in the presence of other enones (Table 3).

Since the insolubility of  $\text{Ba(OAc)}_2$  in  $\text{CH}_3\text{CN}$ , the reaction takes place under heterogeneous conditions. The mechanism for the influence of  $\text{Ba(OAc)}_2$  is not clear. But, we think that with the addition of  $\text{Ba(OAc)}_2$  as Lewis acid to the reaction mixture, the carbonyl group is more susceptible to attack by the hydride ions. Therefore, the rates of reduction reactions have been accelerated.

To highlight the efficiency of our system, we compared our results with other reported reducing systems in the literature such as  $\text{Ph}_3\text{PMeBH}_4$  [21, 22],  $\text{NaBH}_3(\text{OAc})$  [3],  $\text{NaBH}_3\text{CN}$  [23],  $\text{Li-}n\text{-BuBH}_3$  [6],  $(i\text{-PrO})_2\text{TiBH}_4$  [24],  $\text{NaBH}_4/\text{C}$  [25],  $\text{NaBH}_4/\text{wet SiO}_2$  [26],  $\text{NaBH}_4/\text{Dowex1-8x}$  [27], and  $\text{NaBH}_4/\text{DOWEX(R)50WX4}$  [28] (Table 4). Some of the reducing systems have been used in more hydride anions versus one molar equivalent of carbonyl group. Also, it should be noted that the synthesis of some reducing agents is more costly than the  $\text{NaBH}_4/\text{Ba(OAc)}_2$  system. It is clear that in most cases, the  $\text{NaBH}_4/\text{Ba(OAc)}_2$  system is more efficient or comparable in the reaction times and yields of products. Therefore, the  $\text{NaBH}_4/\text{Ba(OAc)}_2$  system has a greater potential for 1,2-reduction of  $\alpha,\beta$ -unsaturated carbonyl compounds.

### 4. Conclusion

In this investigation, we have shown that the combination system of  $\text{NaBH}_4/\text{Ba(OAc)}_2$  in  $\text{CH}_3\text{CN}$  reduces a variety of  $\alpha,\beta$ -unsaturated carbonyl compounds to their corresponding allylic alcohols in high to excellent yields. Reduction reactions were carried out with 0.5–2 molar equivalents of  $\text{NaBH}_4$  in the presence of 0.2 molar amounts of  $\text{Ba(OAc)}_2$ . The chemoselective reduction of enals over enones was accomplished successfully with this reducing system. High efficiency of the reductions, shorter reaction times, and easy work-up procedure make it as an attractive new protocol for reduction of  $\alpha,\beta$ -unsaturated carbonyl compounds, and it could be a useful addition to the present methodologies.

TABLE 1: Optimization of reduction reaction of cinnamaldehyde to 3-phenyl-2-propen-1-ol and benzylideneacetone to 4-phenyl-3-buten-2-ol with  $\text{NaBH}_4/\text{Ba}(\text{OAc})_2$  as a reducing system.

Entry	Substrate	Molar ratio <sup>a</sup>	Solvent	Time (min)	Conversion <sup>b</sup> (%)
1	Cinnamaldehyde	1:0.5:0.5	$\text{CH}_3\text{CN}$	2	100
2	Cinnamaldehyde	1:0.5:0.2	$\text{CH}_3\text{CN}$	5	100
3	Cinnamaldehyde	1:0.25:0.5	$\text{CH}_3\text{CN}$	60	>100
4	Cinnamaldehyde	1:0.5:0.2	THF	10	100
5	Cinnamaldehyde	1:0.5:0.1	$\text{CH}_3\text{CN}$	10	100
6	Benzylideneacetone	1:1:0.5	THF	60	>100
7	Benzylideneacetone	1:1:0.5	$\text{CH}_3\text{CN}$	60	>100
8	Benzylideneacetone	1:2:0.5	$\text{CH}_3\text{CN}$	60	>100
9 <sup>c</sup>	Benzylideneacetone	1:2:0.5	$\text{CH}_3\text{CN}$	15	100
10 <sup>c</sup>	Benzylideneacetone	1:2:0.2	$\text{CH}_3\text{CN}$	20	100

<sup>a</sup>Molar ratio as substrate:  $\text{NaBH}_4$  :  $\text{Ba}(\text{OAc})_2$ ; <sup>b</sup>completion of the reactions was monitored by TLC (eluent; *n*-hexane/EtOAc: 9/1). <sup>c</sup>Reaction was carried out under reflux conditions.

TABLE 2: Reduction of  $\alpha,\beta$ -unsaturated carbonyl compounds with  $\text{NaBH}_4/\text{Ba}(\text{OAc})_2$  as a reducing system in  $\text{CH}_3\text{CN}$ .

Entry	Substrate	Product	Molar ratio <sup>c</sup>	Time (min)	Yield (%) <sup>d</sup>
1 <sup>a</sup>	Cinnamaldehyde	3-phenyl-2-propen-1-ol	1:0.5:0.2	5	94
2 <sup>a</sup>	Citral	3,7-dimethyl-2,6-octadien-1-ol	1:0.5:0.2	5	93
3 <sup>b</sup>	Benzylideneacetone	4-phenyl-3-buten-2-ol	1:2:0.2	15	95
4 <sup>b</sup>	Chalcone	1,3-diphenyl-2-propen-1-ol	1:2:0.2	15	96
5 <sup>b</sup>	$\beta$ -ionone	4-(2,6,6-trimethylcyclohex-1-enyl)-3-buten-2-ol	1:2:0.2	10	97
6 <sup>b</sup>	2-cyclohexenone	2-cyclohexenol	1:2:0.2	10	95
7 <sup>b</sup>	3-methyl-2-cyclopentenone	3-methyl-2-cyclopentenol	1:2:0.2	10	98

<sup>a</sup>The reduction reaction was carried out at room temperature; <sup>b</sup>the reduction reaction was carried out under reflux conditions. <sup>c</sup>Molar ratio as substrate:  $\text{NaBH}_4$  :  $\text{Ba}(\text{OAc})_2$ ; <sup>d</sup>yields refer to isolated pure products.

TABLE 3: Competitive reduction of cinnamaldehyde with enones by  $\text{NaBH}_4/\text{Ba}(\text{OAc})_2$  as a reducing system at room temperature in  $\text{CH}_3\text{CN}$ .

Entry	Enone	Molar ratio <sup>a</sup>	Time (min)	1,2-reduction of cinnamaldehyde/enone (%) <sup>b</sup>
1	Benzylideneacetone	1:1:0.5:0.2	5	100:0
2	Chalcone	1:1:0.5:0.2	5	100:0
3	$\beta$ -ionone	1:1:0.5:0.2	5	100:0
4	2-cyclohexenone	1:1:0.5:0.2	5	100:0
5	3-methyl-2-cyclopentenone	1:1:0.5:0.2	5	100:0

<sup>a</sup>Molar ratio as substrate: cinnamaldehyde : enone :  $\text{NaBH}_4$  :  $\text{Ba}(\text{OAc})_2$ ; <sup>b</sup>completion of the reactions was monitored by TLC (eluent; *n*-hexane/EtOAc: 9/1).

TABLE 4: Comparison of regioselective 1,2-reduction of  $\alpha,\beta$ -unsaturated carbonyl compounds with  $\text{NaBH}_4/\text{Ba}(\text{OAc})_2$  system and other reported systems.

Entry	Reducing systems	Molar ratio, <sup>a</sup> time (h), and yield (%)					
		Cinnamaldehyde	Citral	Benzylideneacetone	Chalcone	$\beta$ -ionone	2-cyclohexenone
1	$\text{NaBH}_4/\text{Ba}(\text{OAc})_2$	(0.5)(0.08)(94)	(0.5)(0.08)(93)	(2)(0.25)(95)	(2)(0.25)(96)	(2)(0.25)(97)	(2)(0.25)(95)
2	$\text{Ph}_3\text{PMeBH}_4$	(1)(Im)(95)	—	(1)(3.5)(90)	(1.2)(6)(90)	(1)(6)(71)	—
3	$\text{NaBH}_3(\text{OAc})$	(1.67)(20)(70)	(1.67)(20)(70)	(1.67)(20)(86)	—	—	(1.67)(20)(32)
4	$\text{NaBH}_3\text{CN}$	(2)(90)(80)	—	(2)(90)(77)	(3)(150)(0)	(2)(120)(88)	—
5	$\text{Li-}n\text{-BuBH}_3$	—	—	(1)(2)(98)	(2)(2)(99)	(1)(2)(98)	(1)(2)(84)
6	( <i>i</i> -PrO) <sub>2</sub> TiBH <sub>4</sub>	(1)(0.08)(90)	(1)(0.08)(95)	—	—	—	—
7	$\text{NaBH}_4/\text{C}$	(0.5)(Im)(92)	(0.5)(0.16)(92)	(2)(0.67)(96)	(2)(0.4)(98)	(2)(0.5)(91)	—
8	$\text{NaBH}_4/\text{wet SiO}_2$	(1)(0.016)(96)	(1)(0.066)(95)	(2)(0.15)(97)	(2)(0.25)(98)	(2)(0.25)(97)	—
9	$\text{NaBH}_4/\text{Dowex1-8x}$	(1)(0.7)(96)	(1)(1.3)(94)	(1)(1.4)(98)	(1)(0.7)(95)	(1)(2.2)(91)	(1)(0.8)(89)
10	$\text{NaBH}_4/\text{DOWEX(R)50WX4}$	(1)(0.25)(97)	(1)(0.33)(94)	(2)(0.91)(95)	(2)(1.5)(95)	—	—

<sup>a</sup>Reducing agent/substrate. Im: immediately.

## Acknowledgment

The authors gratefully appreciated the financial support of this work by the research council of Islamic Azad University, branch of Mahabad.

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