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Baker's yeast mediated asymmetric reduction of cinnamaldehyde derivatives

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Abstract

The enantioselective reduction of cinnamaldehyde derivatives is an attractive strategy to prepare various optically active multifunctional molecules that can be used as chiral building blocks for the synthesis of some HIV-protease inhibitors. The asymmetric reduction with pH adjusted to 5.5 of α-substituted-cinnamaldehydes (Br, N_3) mediated by baker's yeast (*Saccharomyces cerevisiae*) yielded α-substituted-3-phenyl-1-propanol in excellent enantiomeric excesses and yields.

Keywords: HIV-protease inhibitors; Baker's yeast; α-Substituted-3-phenyl-1-propanol

1. Introduction

L-phenylalaninol **1** has been used as chiral auxiliary [1] and is an important intermediate for the synthesis of some HIV-1-protease inhibitors [2] that have been approved by the FDA as a therapeutic agent for treatment of AIDS [3]. Generally, the aminoacid L-phenylalanine **2** is used as raw material for preparation of **1**, which in some routes, is oxidised to aldehyde **3** and then transformed into epoxide **4** (Scheme 1).

The bioreduction of C=C and C=O conjugated double bonds is an important method for preparation of chiral building blocks. It has been successfully achieved in enones by baker's yeast [4] and the stereoselective reduction of tris-substituted C=C double bonds of α,β-unsaturated aldehydes, mediated by baker's yeast, have been extensively studied by Fuganti and coworkers [5].

In this work, we describe an alternative enantioselective synthesis of L- and D-phenylalaninol using cinnamaldehyde as starting material (Scheme 2). The key step of our strategy is the preparation of compound **1** by highly enantioselective reduction of α-substituted cinnamaldehyde mediated by baker's yeast.

2. Results and discussion

The α-substituted cinnamaldehydes **5** and **7** were obtained in good yields from cinnamaldehyde following methods described by Cromwel [6] and Hassner et al. [7] respectively. The reduction of **7** by baker's yeast was performed by stirring the reaction mixture at 30 °C. We observed that alcohols **8** and **9** were obtained as reaction products from C=C and/or C=O bond reductions (Scheme 3). In order to optimise the yield and e.e. of alcohol **8**, a time-course study was developed to determine the influence of sugar and $CaCO_3$ additions and the pH of reaction mixture on the reaction products. Therefore, samples were withdrawn from the reaction mixture, at appropriate intervals of time, and analysed by GC/MS technique in order to determine the relative rates of these bonds reduction.

The reaction profile of fermenting baker's yeast reduction of **7** (with addition of sugar to the reaction mixture) is presented in Fig. 1a. The aldehyde **7** was totally consumed in less than 1 h of reaction while the allyl alcohol **9** and the halohydrine **8** were formed along with a secondary product that seems to be the diol **11**. Diols of type **11** (from acyloin condensation of **7** with acetaldehyde) have been frequently found in the bioreduction of 2-methylcinnamaldehyde **12** affording (*S*)-2-methyl-1-phenyl-1-propanol **13** and the diol **14** [5c]

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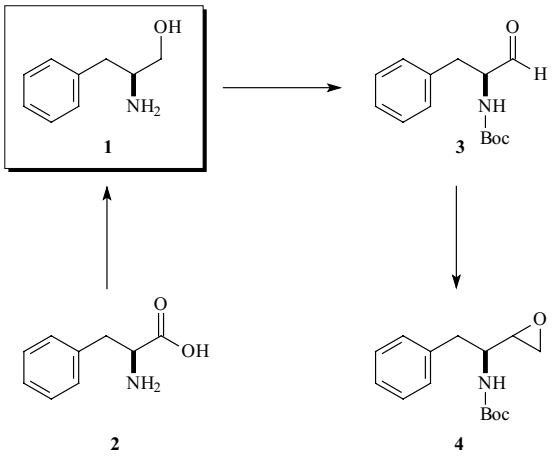
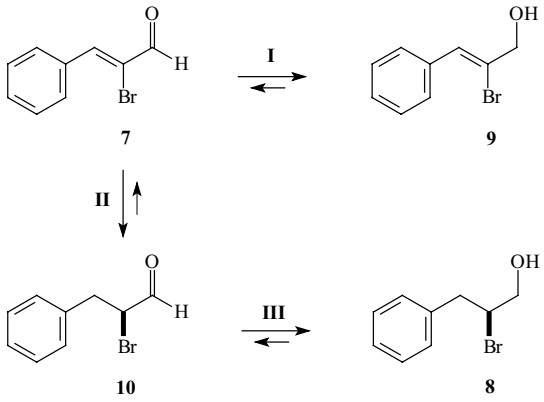
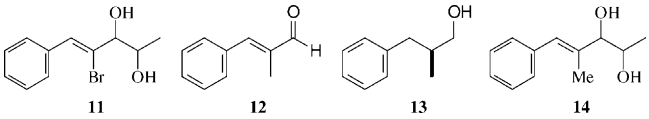


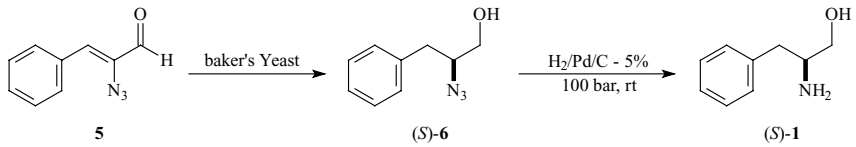
Scheme 1.

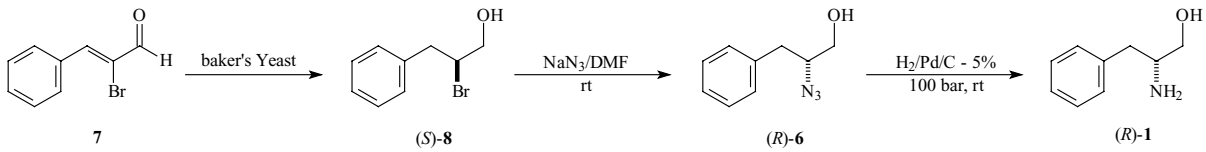


Scheme 3.

A reaction sequence depicted in Scheme 3 was proposed by Fuganti and coworkers, based on experiments with deuterated substrates [5c] may be used to explain the obtained reactions profiles. The equilibrium of step **I** was initially attained favouring to product **9** and the equilibrium







Scheme 2.

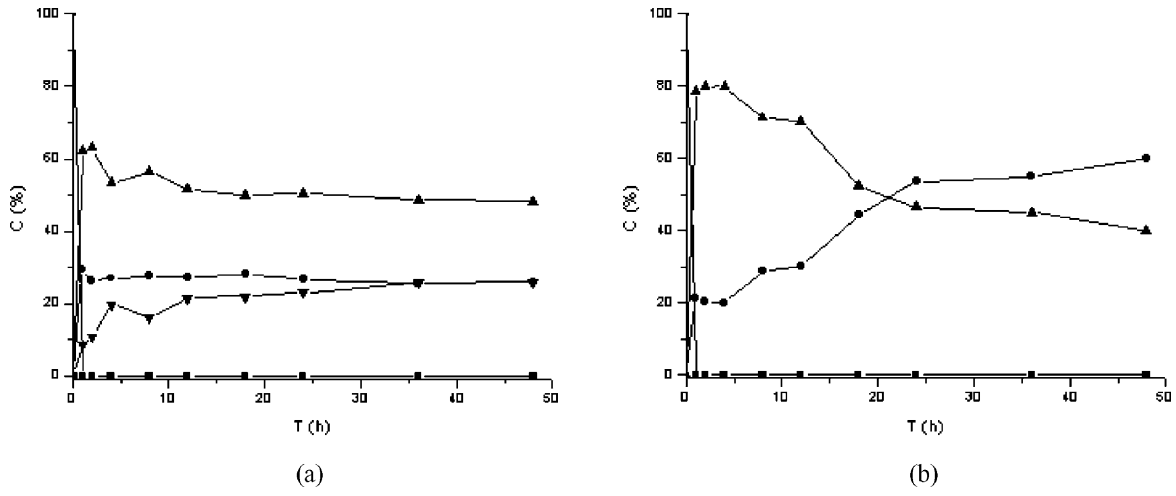


Fig. 1. Reduction of 2-bromo-3-phenyl-2-propenal (■) by baker's yeast affording (*S*)-2-bromo-3-phenyl-1-propanol (●); 2-bromo-3-phenyl-2-propen-1-ol (▲) and 4-bromo-5-phenyl-4-pentene-2,3-diol (▼). (a) Fermenting baker's yeast reduction: $T = 30\,^{\circ}C$, 48 h, 4.7 mmol/25 g (aldehyde/baker's yeast), 10.4 g of glucose, 250 ml H_2O (2.0 ml of EtOH). (b) Non-Fermenting baker's yeast reduction: $T = 30\,^{\circ}C$, 48 h, 4.7 mmol/25 g (aldehyde/baker's yeast), 250 ml H_2O (2.0 ml of EtOH).

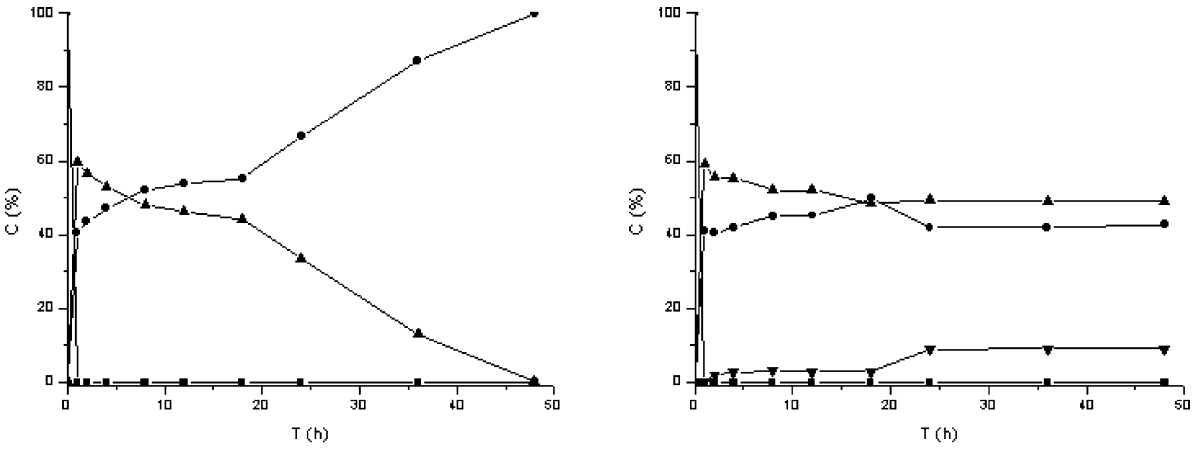


Fig. 2. Reduction of 2-bromo-3-phenyl-2-propenal (■) by baker's yeast affording (*S*)-2-bromo-3-phenyl-1-propanol (●); 2-bromo-3-phenyl-2-propen-1-ol (▲) and 4-bromo-5-phenyl-4-pentene-2,3-diol (▼). (a) Fermenting baker's yeast reduction: $T = 30\,^{\circ}C$, 48 h, pH = 5.5 (calcium carbonate), 4.7 mmol/25 g (aldehyde/baker's yeast), 10.4 g of glucose, 250 ml H_2O (2.0 ml of EtOH). (b) Fermenting baker's yeast reduction: $T = 30\,^{\circ}C$, 48 h, pH = 5.5 (phosphate buffer), 4.7 mmol/25 g (aldehyde/baker's yeast), 10.4 g of glucose, 250 ml H_2O (2.0 ml of EtOH).

Table 1
Reduction of cinnamaldehydes **5**, **7** and **12** by baker's yeast[a]

Aldehyde	Alcohol	Yield (%)	$[\alpha]_D^{20}$	e.e.[b] (%)	Configuration
5	**6**	98	−7.6 (ca. 0.75, $CHCl_3$)	>99	(*S*)
7	**8**	98	−22.5 (ca. 5, $CHCl_3$)	>99	(*S*)
12	**13**	99	−11.0 (ca. 4.6, benzene)	>99	(*S*)

[a] $T = 30\,^{\circ}C$, 48 h, pH = 5.5, 4.7 mmol/25 g (aldehyde/baker's yeast), 10.4 g of glucose, 250 ml H_2O (2.0 ml of EtOH).
[b] Determined by GC–MS analysis (capillary chiral column CHIRASIL-DEX).

of steps **II** and **III** were also initially attained producing **8**. No accumulation of **10** was observed and the mixture of products **8** and **9** remain almost constant up to 48 h of reaction.

The profile of the baker's yeast reduction of **7** (without addition of sugar to the reaction mixture, non-fermenting condition) provided valuable information (Fig. 1b). The aldehyde **7** was also totally consumed in less than 1 h of reaction while the allyl alcohol **9** and the halohydrine **8** were formed without the appearance of diol **11**. In this profile, the allyl alcohol **9** is rapidly formed and its concentration decrease with the reaction time while the concentration of halohydrine **8** increases, but after 48 h, a mixture of products **8** and **9** persists in the reaction media. It seems that the equilibrium positions of the reactions were modified, in a way that the initially formed compound **9** was gradually been converted in **8** through the equilibriums **I**–**III**.

An excellent result was obtained when the fermenting baker's yeast reduction of **7** was performed with the pH of the reaction mixture adjusted to 5.5 and with the addition of calcium carbonate, as shown in Fig. 2a. The reaction profile is almost like those depicted in Fig. 1b, but in this case only the halohydrine **8** is obtained after 48 h of reaction. While the use of phosphate buffer also gave acyloin condensation **7** with acetaldehyde (see Fig. 2b), calcium carbonate seems to modify more effectively the equilibrium positions of reactions in favour of product **8** in a way that the compound **9** initially formed was completely converted in **8** through the equilibriums **I**–**III** in 48 h of reaction.

Therefore, we use the pH adjust to 5.5 with addition of $CaCO_3$ for the fermenting baker's yeast reduction of **5** and **7** in order to prepare compounds (*S*)-**6** and (*S*)-**8**, respectively, with high yields and e.e. (see Table 1). Also, excellent results were obtained in the reduction of 2-methylcinnamaldehyde **12** affording (*S*)-2-methyl-1-phenyl-1-propanol **13**.

3. Conclusion

Bioreduction of α-bromo-, α-azido- and α-methylcinnamaldehyde mediated by baker's yeast gave (*S*)-2-bromo-, (*S*)-2-azido- and (*S*)-2-methyl-3-phenyl-1-propanol in excellent enantiomeric excesses and yields. The optimum conditions were pH adjusted to 5.5 and the presence of calcium carbonate, which resulted in a high performance of the yeast and avoided the parallel acyloin condensation competition.

4. Experimental

IR spectra were recorded on a Bomem MB Series spectrometer. ^1H and ^{13}C NMR spectra were recorded

on a Varian Gemini 300 spectrometer using $CDCl_3$ as solvent. The melting points were obtained on MQAPF-301-MicroQuímica equipment. The gas chromatographic analysis were performed using a Shimadzu GC/MS Class 5000, with helium as carrier gas, with fused silica capillary columns of SUPELCO SIMPLICITY 1^{TM} (30 m × 0.25 mm × 0.25 μm) and CHIRASIL-DEX (25 m × 0.25 mm × 0.25 μm). Optical rotations were measured with a J-720 VRDM306 JASCO spectropolarimeter (589.3 nm, 25 °C). Cinnamaldehyde was acquired from the Aldrich Co. All other solvents and reagents were reagent grade. The commercially available dry baker's yeast Emulzint® was used in this work [8].

Racemic **6** was necessary for e.e. GC determination and was obtained by reacting NaN_3/DMF with racemic **8** that was prepared from 2-phenylpropene following a methodology published elsewhere [9]. The optically active **6**, obtained by baker's yeast reduction of **5**, was quantitatively reduced by H_2/Pd/C to (*S*)-**1** for determination of its configuration ($[\alpha]_D^{25}$ − 22.6° (ca. 1.2, HCl 1 M); lit.$[\alpha]_D^{25}$ − 22.8° (ca. 1.2, HCl 1 M, >99% e.e. (*S*)) [10]. In addition, the bromohydrine (*S*)-**8** was treated with NaN_3/DMF to give (*R*)-**6** that was reduced by H_2/Pd/C to (*R*)-**1**.

4.1. General procedure for bioreduction of α-substituted-cinnamaldehydes

Respective α-substituted-cinnamaldehyde (4.7 mmol) dissolved in 1.5 ml of ethanol was added to a mixture of 25 g dry baker's yeast (Emulzint®) and 10.4 g of glucose in 250 ml of water at 30 °C and pH = 5.5 (the pH of the fermenting yeast mixture was adjusted to 5.5 by the addition of saturated sodium carbonate solution). The resulting suspension was stirred in an orbital shaker (200 rpm) at 30 °C until full conversion (48 h). The product was extracted with CH_2Cl_2 and purified by column chromatography using hexane/ethyl acetate (7:3).

4.2. 2-Azido-3-phenyl-2-propenal (5)

To a stirred slurry of 1.2 g (18.5 mmol) of sodium azide in 20 ml of acetonitrile cooled methanol-dry ice bath, 3.0 g (18.5 mmol) of iodine monochloride in 10 ml of acetonitrile was slowly added over a period of 10 min. The reaction mixture was stirred for an additional 10 min. followed by addition of 2.3 g (18.2 mmol) of the cinnamaldehyde, and then allowed to warm to room temperature and stirred for 6 h. The red-brown slurry was poured into 100 ml of water, the adduct was extracted with 200 ml of ether and washed with 300 ml of 5% sodium thiosulfate aqueous solution in three portions leaving a colourless solution. The organic phase was dried on $MgSO_4$. Evaporation of the solvent furnished crude azide adduct (slightly orange) and used in the reaction of preparation of **5**.

A solution of 5.0 g (16.6 mmol) of azide adduct (3-azido-2-iodo-3-phenylpropanal) and 4.0 g (61.5 mmol) of sodium azide in 30 ml of DMF (dried over molecular sieves, type 4A) was stirred for 1 h at room temperature. The solution was then poured into a mixture of water–ether, and the ether layer was washed with water and dried ($MgSO_4$). Evaporation of the solvent furnished crude aldehyde **5**. Purification was achieved by flash column chromatography using hexane/ethyl acetate (7:3), affording **5**, a yellow crystalline solid, mp 73–74 °C, 96% yield.

IR (KBr) (cm^{-1}) 2956, 2923, 2868, 2134, 1674, 1611, 1449, 1404, 1368, 1324, 1299, 1260, 1150, 856, 836, 769, 750, 690. ^{1}H NMR (300 MHz, $CDCl_3$): δ 6.48 (s, 1H); 7.40–7.46 (m, 3H); 7.85–7.89 (m, 2H); 9.46 (s, 1H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 128.53; 130.31; 130.62; 131.08; 132.62; 134.48; 188.14. MS *m/z* (%) 145 (16), 132 (48), 131 (69), 118 (6), 117 (13), 103 (46), 90 (26), 77 (63), 51 (100), 40 (71).

4.3. (S)-2-Azido-3-phenyl-1-propanol ((S)-6)

When 0.8 g (4.7 mmol) of 2-azido-3-phenylpropenal **5** was subjected to the general procedure for bioreduction, the isolated product was (*S*)-**6** (0.8 g, 97.7%) as a yellow oil; $[\alpha]_D^{25}$ − 7.6° (ca. 0.75, $CHCl_3$); IR (film) (cm^{-1}) 3362, 3064, 3028, 2926, 2876, 2112, 1645, 1496, 1451, 1264, 1030, 749, 697. ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.85 (sl, 1H); 2.83 (dd, 1H, J = 7.0, 13.6 Hz); 2.90 (dd, 1H, J = 6.6, 13.6 Hz); 3.55–3.68 (m, 1H); 3.70 (dd, 1H, J = 7.3, 13.6 Hz); 3.73 (dd, 1 H, J = 5.0, 13.6 Hz); 7.22–7.37 (m, 5H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 36.95; 64.31; 65.19; 126.70; 128.46; 129.02; 136.73. MS *m/z* (%) 119 (2) 118 (13), 92 (14), 91 (100), 77 (4), 65 (24).

4.4. 2-Bromo-3-phenyl-2-propenal (7)

A solution of 5.0 g (37.88 mmol) of cinnamaldehyde in 15 ml of CCl_4 was cooled to 0 °C and treated dropwise with 6.1 g (38.1 mmol) of bromine in 5 ml of CCl_4. The mixture was stirred vigorously during 1 h, washed with a aqueous solution of sodium bisulfite and the organic phase was dried on $MgSO_4$. Evaporation of the solvent furnished crude aldehyde **7**. Purification was achieved by flash column chromatography using hexane/ethyl acetate (7:3), affording **7**, a yellow crystalline solid, mp 72 °C, 87.6% yield.

IR (KBr) (cm^{-1}) 2959, 2924, 2851, 2834, 1687, 1666, 1603, 1448, 1291, 1117, 880, 762, 686, 546, 520. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.47–7.5 (m, 3H); 7.89 (s, 1H); 7.97–8.0 (m, 2H); 9.3 (s, 1H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 124.37; 128.95; 131.15; 131.79; 133.08; 149.58; 187.39. MS *m/z* (%) 212 (25), 211 (38), 210 (26), 209 (37), 184 (2), 182 (2), 131 (25), 130 (7), 103 (86),.

4.5. (S)-2-Bromo-3-phenyl-1-propanol (S)-8

When 1.0 g (4.7 mmol) of 2-bromo-3-phenyl-2-propenal **7** was subjected to the general procedure for bioreduction, the isolated product was (*S*)-**8** (1.0 g, 98.1%) as a yellow oil;

$[\alpha]_D^{25} - 22.5^\circ$ (ca. 5, $CHCl_3$) ($[\alpha]_D^{25} - 22.6^\circ$ (ca. 5, $CHCl_3$), >99% e.e. (*S*)); [5d,e] IR (film) (cm^{-1}) 3372, 3086, 3062, 3027, 3002, 2926, 2858, 1495, 1453, 1042, 1031, 967, 749, 699. ^1H NMR (300 MHz, $CDCl_3$): δ 2.15 (sl, 1 H); 3.15 (dd, 1H, $J = 7.3, 14.0$ Hz); 3.25 (dd, 1H, $J = 7.3, 14.0$ Hz); 3.80 (m, 2H); 4.27 (m, 1H); 7.30 (m, 5H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 41.27; 58.60; 65.91; 126.80; 128.37; 128.95; 137.41. MS *m*/*z* (%) 216 (1), 214 (1), 134 (10), 118 (8), 117 (86), 92 (23), 91 (100).

*4.6. (S)-2-Methyl-3-phenyl-1-propanol (S)-**13***

When 0.7 g (4.7 mmol) of 2-azido-3-phenylpropenal **12** was subjected to the general procedure for bioreduction, the isolated product was (*S*)-**13** (0.7 g, 99%) as a yellow oil; $[\alpha]_D^{25} - 11.6^\circ$ (ca. 4.6, benzene) ($[\alpha]_D^{25} - 11.08^\circ$ (ca. 4.6, benzene), >99% e.e. (*S*)) [11] IR (film) (cm^{-1}) 3345, 3027, 2957, 2923, 2872, 1602, 1495, 1032, 739, 700. ^1H NMR (300 MHz, $CDCl_3$): δ 0.92 (d, 3H, $J = 7$ Hz); 1,35 (sl, 1H); 1.96 (m, 1H); 2.43 (dd, 1H, $J = 8.0, 13.2$ Hz); 2.75 (dd, 1H, $J = 6.2, 13.2$ Hz); 3.47 (dd, 1H, $J = 5.9, 10.6$ Hz); 3.54 (dd, 1H, $J = 5.9, 10.6$ Hz); 7.15–7.21 (m, 3H); 7.25–7.31 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 16.44; 37.73; 39.64; 67.55; 125.65; 128.03; 128.90; 140.36. MS *m*/*z* (%) 150 (7), 132 (11), 117 (44), 115 (7), 104 (3), 92 (44), 91 (100).

Acknowledgements

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