SIMPLE AND EFFICIENT SYNTHESIS OF NEWBENZO[4,5]IMIDAZO[1,2-A]PYRIMIDINE DERIVATIVES USING ACETIC ACID AS CATALYST IN ETHANOL MEDIUM

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ABSTRACT
A series of new 2-(1H-benzo[d]imidazol-2-yl)-4-phenylbenzo[4,5]imidazo[1,2-a]pyrimidine derivatives 6a-g were synthesized by simple condensation reaction between 1-(1H-benzo[d]imidazol-2-yl)-3-phenylprop-2-en-1-one derivatives 5a-g and 2-aminobenzimidazole in the presence of catalytic amount of acetic in ethanol are heated under reflux for 2-3 hours. The yield of the synthesized compounds varied from 89-94%. The structures of the compounds obtained were characterized and confirmed by IR, 1H-NMR, 13C-NMR.

INTRODUCTION
During the last decades, chemists have been interesting on heterocyclic compounds and their various derivatives, especially the one that contains nitrogen, considering that it's excite in nucleic acids, vitamins, proteins and important molecular systems as well as their applications in pharmaceutical and chemical fields. Benzimidazole and pyrimidine are one of these heterocyclic aromatic organic compounds containing nitrogen. The benzimidazole ring system has been found to be an integral part of Vitamin-B12 and in the form of 5,6-dimethyl-1-(α-D-ribofuranosyl) benzimidazole. The pyrimidine ring system has wide occurrence in nature as substituted and ring fused compounds and derivatives, including the nucleotides cytosine, thymine and uracil, thiamine (vitamin B1) and alloxan. Over the past years, several Pyrimidobenzimidazoles derivatives have been synthesized and widely screened for their biological activities. These classes of heterocycles have found applications in diverse pharmacological areas such as anticancer, antiviral, anti-inflammatory, and others.
purity of the synthesized compounds was monitored by TLC and the structures of all precursor benzimidazolyl-N2-phenylbenzo[4,5]imidazo[1,2-a]pyrimidine derivatives were characterized and confirmed by IR, 1H NMR, 13C-NMR.

RESULTS AND DISCUSSION
In this work, a simple and practical method for the preparation of new 2-(1H-benzo[d]imidazol-2-yl)-4-phenylbenzo[4,5]imidazo[1,2-a]pyrimidine derivatives through the condensation reaction of 1-(1H-benzo[d]imidazol-2-yl)-3-phenylprop-2-en-1-ones with ON-phenylenediamine in ethanol are heated under reflux for 2-3 hours (Table 2). The reaction conditions were optimized by conducting the reaction model at different solvents such as methanol, water, ethanol, DMF, Butanol and also under solvent-free condition (Table 1). The intermediates were prepared by condensing the ON-phenylenediamine with the proposed structures.

The precursors were prepared by condensing the ON-phenylenediamine with lactic acid in HCl 4N using the condensation of Phillip method. An oxidation has been carried out on the precursors by potassium dichromate in the sulfuric acid medium leading to the formation of 2-acetyl benzimidazole. The synthetic pathway compounds are provided in (Scheme 01). The purity of the synthesized compounds was monitored by TLC and the structures of all compounds were supported by spectral data. The IR, 1H NMR and 13C-NMR are consistent with the proposed structures.

Scheme 01: synthetic route for the preparation of the new 2-(1H-benzo[d]imidazol-2-yl)-4-phenyl-3,4-dihydrobenzo[4,5]imidazo[1,2-a]pyrimidine 6a-g
Table 1: The synthesis of 6a-g compounds by using different solvents.

<table>
<thead>
<tr>
<th>Entry</th>
<th>Catalyst (0.5eq)</th>
<th>Solvent</th>
<th>Time (min)</th>
<th>Temp (°C)</th>
<th>Yielda (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>AcOH</td>
<td>Free Solvent</td>
<td>24</td>
<td>80</td>
<td>75</td>
</tr>
<tr>
<td>2</td>
<td>AcOH</td>
<td>Ethanol</td>
<td>3h</td>
<td>Reflux</td>
<td>94</td>
</tr>
<tr>
<td>3</td>
<td>AcOH</td>
<td>Methanol</td>
<td>5</td>
<td>Reflux</td>
<td>85</td>
</tr>
<tr>
<td>4</td>
<td>AcOH</td>
<td>Chloroform</td>
<td>24</td>
<td>Reflux</td>
<td>-</td>
</tr>
<tr>
<td>5</td>
<td>AcOH</td>
<td>DMF</td>
<td>12</td>
<td>Reflux</td>
<td>67</td>
</tr>
<tr>
<td>6</td>
<td>AcOH</td>
<td>Butanol</td>
<td>24</td>
<td>Reflux</td>
<td>81</td>
</tr>
</tbody>
</table>

Table 3: Synthesis of products 9a-g by the reactions of 5a-g with 2-aminobenzimidazole in the presence of AcOH (0.52mmol), at Reflux in Ethanol.

<table>
<thead>
<tr>
<th>R</th>
<th>Product</th>
<th>Time (h)</th>
<th>Yieldb (%)</th>
<th>M.P (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5aH</td>
<td>6a</td>
<td>3</td>
<td>92</td>
<td>234–236</td>
</tr>
<tr>
<td>5</td>
<td>6b</td>
<td>3</td>
<td>94</td>
<td>222–224</td>
</tr>
<tr>
<td>3</td>
<td>6c</td>
<td>2.5</td>
<td>93</td>
<td>236–237</td>
</tr>
<tr>
<td>4</td>
<td>6d</td>
<td>2</td>
<td>92</td>
<td>226-227</td>
</tr>
<tr>
<td>5</td>
<td>6e</td>
<td>3</td>
<td>91</td>
<td>232–233</td>
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<tr>
<td>6</td>
<td>6f</td>
<td>2</td>
<td>93</td>
<td>224-225</td>
</tr>
<tr>
<td>7</td>
<td>6g</td>
<td>1.5</td>
<td>89</td>
<td>245–246</td>
</tr>
</tbody>
</table>

a. Reaction conditions: 5a (1 mmol), 2-aminobenzimidazole (1 mmol) in the presence of AcOH (0.52mmol), at Reflux in Ethanol.
b. Isolated yield

In IR spectra, an absorption band 3248-3291 cm⁻¹ showing the existence of the NH bond in all compounds 6a-g which. The C=N stretching absorption for benzimidazole compounds appeared nearly around 1 cm⁻¹, and absorption region of the C=C stretching at 1470 cm⁻¹, The absorption bands of α,β-unsaturated carbonyl system was absent in the spectra of compounds 6a-g due to the reaction with 2-aminobenzimidazole.

In the result of ¹H RMN, the signal of NH displayed at ~11.5 ppm in the spectra of compounds 6a-g, corresponding at benzimidazole and we observed two peaks, the first as a triplet at 5.08 for the CH of pyrimidine ring and the second represented as doublets at 2.9 for CH2 of pyrimidol[1,2-a]benzimidazole. The protons of α,β-unsaturated system was absent due to the reaction with 2-aminobenzimidazole. Other protons appeared in the expected region.
13C NMR confirmed the suggested structure of compounds 6a-g, by the absence of signals of carbonyl carbon atoms of the chalcone fragment, while the appearance of two signals of carbons of the pyrimidol[1,2-a]benzimidazole in the products of 6a-g, the first at ~34 ppm was assigned to CH₂ group and the second at ~55 ppm was assigned to CH.

CONCLUSION
In summary, using simple condensation reaction between 1-(1H-benzo[d]imidazol-2-yl)-3-phenylprop-2-en-1-one derivatives and 2-aminobenzimidazole in the presence of catalytic amount of acetic in ethanol are heated under reflux, Series of 2-(1H-benzo[d]imidazol-2-yl)-4-phenylbenzo[4,5]imidazo[1,2-a]pyrimidine derivatives were synthesized. The synthesized compounds were obtained in excellent yields and identified by melting point and characterized by IR, 1H NMR and 13C RMN.

EXPERIMENTAL SECTION
Most of the materials used in this work are Sigma Aldrich brand commercial products and have been used without further purification. The melting points were taken by using a Kofler bench melting point apparatus. The purity of the synthesized compounds was checked by TLC using silica gel-60 F 254 aluminium sheets using Ethyl acetate: Petroleum ether (3:7) as eluent and visualized in a ultra violet chamber. IR spectra were recorded on ALPHA's Platinum ATR single reflection diamond ATR spectrophotometer. The 1H NMR and 13C NMR spectra were recorded on a Bruker AC 300 MHZ FTNMR spectrophotometer in CDCl₃ and
DMSO. Chemical shift were recorded in parts per million (ppm) downfield from TMS as an internal reference and the following multiplicity abbreviations were used: s, singlet; d, doublet; t, triplet; q, quadruplet; m, multiplet. All chemicals were obtained from Merck and were used without further purification.

General procedure for synthesis of 1-(1H-benzo[d]imidazol-2-yl)-3-phenylprop-2-en-1-one derivatives (5a-g): A solution of 2-N-acetyl-N-benzimidazole (3.2 g, 20 mmol) and the diversely substituted aromatic aldehyde (24 mmol) in 40 ml of absolute methanol were taken in a flask. 0.2 mmol of Piperidine was added and the reaction mixture was irradiated in a domestic microwave oven for 1 minute to 3 minutes at the power of 360 watts, the reaction is followed by thin layer chromatography using silica gel. The reaction mixture cooled to room temperature. Then poured onto crushed ice water. The product obtained is filtered and washed with distilled water. The compounds obtained recrystallized in suitable solvent.

1-(1H-benzo[d]imidazol-2-yl)-3-phenylprop-2-en-1-one (5a): Yield (93%). Pale yellow crystals. Mp 196–197°C. IR spectrum, ν, cm⁻¹: 3241 (N–H), 1656 (C=O), 1592 (C=N).¹H NMR (300 MHz, DMSO-d₆) δ 13.49 (s, 1H, NH), 8.13 (d, J = 16.1 Hz, 1H), 7.98 (d, J = 16.1 Hz, 1H), 7.86 (d, J = 2.9 Hz, 2H), 7.73 (s, 2H), 7.49 (d, J = 3.0 Hz, 3H), 7.37 (dd, J = 6.0, 3.0 Hz, 2H). ¹³C NMR (75 MHz, DMSO-d₆) δ 180.96, 148.98, 144.21, 134.29, 131.06, 129.11, 128.88, 124.42, 121.58.

1-(1H-benzo[d]imidazol-2-yl)-3-(4-methoxyphenyl)prop-2-en-1-one (5b): Yield (83%). Yellow crystals. Mp 185–187°C IR spectrum, ν, cm⁻¹: 3253 (N–H), 1651 (C=O),1573 (C=N), ¹H NMR (300 MHz, ) δ 13.48 (s, 1H, NH); 8.03 (d, J = 15.9, 1H); 7.96 (d, J = 15.9, 1H); 7.76 (t, J = 14.5 Hz, 2H), 7.59 (d, J = 7.3 Hz, 1H), 7.5 5 – 7.42 (m, 2H), 7.38 (d, J = 2.9 Hz, 1H), 7.36 (d, J = 3.0 Hz, 1H). 3.79 (s, 3H).

13C NMR (75 MHz, DMSO-d₆) δ 181.48, 162.30, 149.11, 145.93, 143.72, 133.85, 131.11, 127.41, 126.29, 123.74, 121.84, 118.63, 114.51, 112.15, 55.45.

1-(1H-benzo[d]imidazol-2-yl)-3-(4-chlorophenyl)prop-2-en-1-one (5c): Yield (89%). Yellow crystals. Mp 201–202°C. IR spectrum, ν, cm⁻¹: 3261 (N–H), 1660 (C=O), 1594 (C=N). ¹H NMR (300 MHz, DMSO-d₆) δ 13.48 (s, 1H, NH), 8.12 (d, J = 16.1 Hz, 1H), 7.94 (d, J = 16.2 Hz, 1H), 7.87 (d, J = 8.4 Hz, 2H), 7.72 (s, 2H), 7.51 (d, J = 8.4 Hz, 2H), 7.36 (d, J = 3.1 Hz, 1H), 7.34 (d, J = 3.1 Hz, 1H). ¹³C NMR (75 MHz, DMSO-d₆) δ 181.07, 149.14, 142.88, 133.47, 130.72, 129.34, 126.63, 124.50.

1-(1H-benzo[d]imidazol-2-yl)-3-(4-bromophenyl)prop-2-en-1-one (5d): Yield (84%). Yellow crystals. Mp 222–223°C. IR spectrum, ν, cm⁻¹: 3249 (N–H), 1659 (C=O), 1596 (C= N). ¹H NMR (300 MHz, DMSO-d₆) δ 13.48 (s, 1H, NH), 8.12 (d, J = 16.1 Hz, 1H), 7.94 (d, J = 16.2 Hz, 1H), 7.87 (d, J = 8.4 Hz, 2H), 7.72 (s, 2H), 7.51 (d, J = 8.4 Hz, 2H), 7.36 (d, J = 3.1 Hz, 1H), 7.34 (d, J = 3.1 Hz, 1H). ¹³C NMR (75 MHz, DMSO-d₆) δ 181.42, 149.47, 133.59, 130.64, 129.49, 131.24, 126.62, 125.01, 122.50.

1-(1H-benzo[d]imidazol-2-yl)-3-(4-nitrophenyl)prop-2-en-1-one (5e): Yield (91%). Yellow crystals. Mp 204–205°C . IR spectrum, ν, cm⁻¹: 3267 (N–H), 1668 (C=O), 1591 (C= N). ¹H NMR (300 MHz, DMSO-d₆) δ 13.78 (s, 1H, NH), 8.70 (d, J = 16.3 Hz, 1H), 8.13 (d, J = 16.2 Hz, 1H), 8.10 (s, 1H), 7.76 (t, J = 14.5 Hz, 2H), 7.59 (d, J = 7.3 Hz, 1H), 7.55 – 7.42 (m, 2H), 7.38 (d, J = 2.9 Hz, 1H), 7.36 (d, J = 3.0 Hz, 1H). ¹³C NMR (75 MHz, DMSO-d₆) δ 181.46, 149.49, 138.31, 134.16, 132.05, 131.21, 130.94, 126.82, 125.01, 122.90.

1-(1H-benzo[d]imidazol-2-yl)-3-(2-chlorophenyl)prop-2-en-1-one (5f): Yield (93%). Yellow crystals. Mp 204–205°C. IR spectrum, ν, cm⁻¹: 3267 (N–H), 1668 (C=O), 1591 (C= N). ¹H NMR (300 MHz, DMSO-d₆) δ 13.52 (s, 1H, NH), 8.26 (d, J = 16.1 Hz, 1H), 8.15 (d, J = 18.1 Hz, 1H), 8.10 (s, 1H), 7.76 (t, J = 14.5 Hz, 2H), 7.59 (d, J = 7.3 Hz, 1H), 7.55 – 7.42 (m, 2H), 7.38 (d, J = 2.9 Hz, 1H), 7.36 (d, J = 3.0 Hz, 1H). ¹³C NMR (75 MHz, DMSO-

1-(1H-benzo[d]imidazol-2-yl)-3-(p-tolyl) prop-2-en-1-one (5g). Yield (86%). Yellow crystals. Mp 195–196°C. IR spectrum, ν, cm⁻¹: 3251 (N–H), 1653 (C=O), 1577 (C=N), 1. H NMR (300 MHz, DMSO-d₆) δ 13.54 (s, 1H, NH); 8.30 (d, J = 16, 1H,); 7.96 (d, J = 7.3 Hz, 1H), 7.55 – 7.42 (m, 2H), 7.38 (d, J = 2.9 Hz, 1H), 7.36 (d, J = 3.0 Hz, 1H). 13C NMR (75 MHz, DMSO-d₆) δ 182.38, 161.30, 149.15, 146.73, 141.72, 133.85, 131.11, 127.41, 126.29, 123.74, 121.84, 118.63, 114.51, 112.15, 55.45.

General procedure for synthesis 2-(1H-benzo[d]imidazol-2-yl)-4-phenylbenzo[4,5]imidazo[1,2-a]pyrimidine (6 a-g):
2-N-aminobenzimidazole (1 mmol) and 5 a-g compounds (1 mmol) were taken in ethanol in a 50 mL round bottomed flask and stirred for 5 min. AcOH (0.5 mmol) was added and the reaction mixture was stirred at reflux for 2–3 h. The progress of the reaction was monitored by TLC. After completion of the reaction, the crude mixture was partitioned between H₂O and EtOAc. The aqueous layer was extracted with EtOAc (3×15 mL). The combined organic phases were dried (MgSO₄) and concentrated under reduced pressure. The compounds obtained recrystallized in ethanol absolute.

2-(1H-benzo[d]imidazol-2-yl)-4-phenylbenzo[4,5]imidazo[1,2-a]pyrimidine (6a): Yield (92%). Yellowish green solid. Mp 234–236°C. IR spectrum, ν, cm⁻¹: 3267 (N–H), 1591 (C=N). 1H NMR (300 MHz, DMSO-d₆) δ 11.06 (s, 1H), 8.13 – 8.04 (m, 1H), 7.74 (dd, J = 1.5, 7.5 Hz, 1H), 7.64 – 7.37 (m, 3H), 7.36 – 7.24 (m, 4H), 7.28 – 7.11 (m, 1H), 7.17 – 7.03 (m, 1H), 7.04 (dd, J = 0.8, 1.6, 6.5 Hz, 1H), 5.08 (t, J = 0.6, 9.1 Hz, 1H), 2.77 (d, J = 9.3 Hz, 2H). 13C NMR (75 MHz, DMSO-d₆) δ 161.43, 150.69, 145.06, 142.41, 141.49, 139.80, 137.58, 135.30, 128.57, 127.79, 125.08, 121.43, 121.31, 121.17, 118.59, 117.11, 113.49, 111.10, 53.59, 32.71.

2-(1H-benzo[d]imidazol-2-yl)-4-(4-methoxyphenyl)benzo[4,5]imidazo[1,2-a]pyrimidine (6b): Yield (94%). Yellowish grey solid. Mp 222–224°C. IR spectrum, ν, cm⁻¹: 3235 (N–H), 1598 (C=N). 1H NMR (300 MHz, DMSO-d₆) δ 11.03 (s, 1H), 7.68 – 7.59 (m, 1H), 7.64 – 7.52 (m, 3H), 7.35 – 7.17 (m, 6H), 6.94 – 6.84 (m, 2H), 5.00 (t, J = 0.6, 9.2 Hz, 1H), 3.81 (s, 3H), 3.02 (d, J = 9.3 Hz, 2H). 13C NMR (75 MHz, DMSO-d₆) δ 161.43, 158.29, 150.69, 145.06, 142.41, 141.49, 137.58, 135.30, 128.57, 127.79, 125.08, 121.43, 121.31, 121.17, 118.59, 117.11, 113.49, 111.10, 53.59, 53.56, 32.13.

2-(1H-benzo[d]imidazol-2-yl)-4-(4-chlorophenyl)benzo[4,5]imidazo[1,2-a]pyrimidine (6c): Yield (93%). Yellowish green solid. Mp 236–237°C. IR spectrum, ν, cm⁻¹: 3259 (N–H), 1589 (C=N). 1H NMR (300 MHz, DMSO-d₆) δ 11.18 (s, 1H), 7.86 – 7.77 (m, 1H), 7.77 – 7.69 (m, 1H), 7.58 (dd, J = 1.5, 7.2 Hz, 1H), 7.40 – 7.11 (m, 7H), 7.04 (dd, J = 0.7, 1.5, 7.6 Hz, 1H), 5.16 – 5.03 (m, 1H), 2.92 (d, J = 9.3 Hz, 2H). 13C NMR (75 MHz, DMSO-d₆) δ 161.43, 150.69, 145.06, 142.41, 141.49, 137.58, 135.30, 134.78, 128.75, 125.08, 121.43, 121.31, 121.17, 118.59, 117.11, 114.14, 113.49, 111.10, 55.40, 53.56, 32.13.

2-(1H-benzo[d]imidazol-2-yl)-4-(4-bromophenyl)benzo[4,5]imidazo[1,2-a]pyrimidine (6d): Yield (92%). Yellowish green solid. Mp 226–227°C. IR spectrum, ν, cm⁻¹: 3271 (N–H), 1584 (C=N). 1H NMR (300 MHz, DMSO-d₆) δ 11.08 (s, 1H), 7.97 – 7.89 (m, 1H), 7.81 – 7.72 (m, 1H), 7.68 – 7.52 (m, 3H), 7.47 (dd, J = 1.5, 7.2 Hz, 1H), 7.35 – 7.22 (m, 3H), 7.21 (dd, J = 1.7, 7.3, 7.8 Hz, 1H), 7.15 – 7.06 (m, 2H), 5.00 (td, J = 0.6, 9.2 Hz, 1H), 3.10 (d, J = 9.3 Hz, 2H). 13C NMR (75 MHz, DMSO-d₆) δ 161.43, 150.69, 145.06, 142.41, 141.49, 138.97, 137.58, 135.30, 130.25, 129.25, 128.53, 125.08, 121.43, 121.31, 121.17, 118.59, 117.11, 113.49, 111.10, 55.28, 34.49.
2-(1H-benzo[d]imidazol-2-yl)-4-(4-nitrophenyl)benzo[4,5]imidazo[1,2-a]pyrimidine (6e): Yield (91%). yellowish green solid. Mp 232–233°C. $^1$H NMR (300 MHz, DMSO-d$_6$) $\delta$ 11.11 (s, 1H), 8.09 (dd, $J$ = 1.5, 7.6 Hz, 1H), 8.00 (dd, $J$ = 1.5, 7.6 Hz, 1H), 7.72 – 7.17 (m, 11H), 5.00 (tt, $J$ = 0.6, 9.2 Hz, 1H), 2.91 (d, $J$ = 9.3 Hz, 2H). $^{13}$C NMR (75 MHz, DMSO-d$_6$) $\delta$ 161.43, 150.69, 146.79, 145.06, 143.06, 142.41, 141.49, 137.58, 135.30, 126.32, 125.08, 124.79, 121.43, 121.31, 121.17, 118.59, 117.11, 113.49, 111.10, 55.75, 33.78.

2-(1H-benzo[d]imidazol-2-yl)-4-(2-chlorophenyl)benzo[4,5]imidazo[1,2-a]pyrimidine (6f): Yield (93%). yellowish brown solid. Mp 224–225°C. IR spectrum, $\nu$, cm$^{-1}$: 3261 (N–H), 1575 (C=N). $^1$H NMR (300 MHz, DMSO-d$_6$) $\delta$ 11.05 (s, 1H), 7.68 – 7.59 (m, 1H), 7.63 – 7.54 (m, 3H), 7.59 – 7.53 (m, 1H), 7.46 (td, $J$ = 1.4, 7.7 Hz, 1H), 7.35 – 7.04 (m, 5H), 6.99 (ddd, $J$ = 1.6, 6.7, 8.0 Hz, 1H), 5.01 (td, $J$ = 0.6, 9.7 Hz, 1H), 2.76 (d, $J$ = 9.6 Hz, 2H). $^{13}$C NMR (75 MHz, DMSO-d$_6$) $\delta$ 161.43, 150.69, 145.06, 142.41, 141.49, 140.83, 137.58, 135.30, 134.74, 131.03, 130.16, 128.79, 126.09, 125.08, 121.43, 121.31, 121.17, 118.59, 117.11, 113.49, 111.10, 53.99, 33.56.

2-(1H-benzo[d]imidazol-2-yl)-4-(p-tolyl)benzo[4,5]imidazo[1,2-a]pyrimidine (6g): Yield (89%). yellowish brown solid. Mp 245–246°C. IR spectrum, $\nu$, cm$^{-1}$: 3263 (N–H), 1585 (C=N). $^1$H NMR (300 MHz, DMSO-d$_6$) $\delta$ 11.08 (s, 1H), 7.68 – 7.52 (m, 4H), 7.45 – 7.24 (m, 3H), 7.29 – 7.02 (m, 6H), 5.09 (t, $J$ = 0.6, 9.1 Hz, 1H), 2.88 (d, $J$ = 9.3 Hz, 2H), 2.30 (s, 3H). $^{13}$C NMR (75 MHz, DMSO-d$_6$) $\delta$ 161.43, 150.69, 145.06, 142.41, 141.49, 137.58, 136.92, 135.30, 129.66, 126.09, 125.08, 121.43, 121.31, 121.17, 118.59, 117.11, 113.49, 111.10, 54.96, 34.80, 18.97.

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