

**REGIOSELECTIVE SYNTHESIS OF NOVEL 1,4 DISUBSTITUTED BIS 1,2,3  
TRIAZOLES : CLICK CHEMISTRY APPROACH**

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**ABSTRACT:**

Regioselective synthesis of a series of new 1,4-disubstituted bis 1,2,3-triazoles **3a-j** from 2,4-bis-propargyloxy acetophenones and organic azides are reported. Optimal experimental conditions were established for these triazoles catalysed by copper (II) sulfate pentahydrate in presence of sodium ascorbate by the conventional click chemistry. The compounds are characterized by the IR, <sup>1</sup>H NMR, MS.

**KEYWORDS:** 1,2,3-Triazoles, Regioselective synthesis, propargyloxy acetophenones, organic azides, click chemistry

**INTRODUCTION:**

1,2,3-Triazole compounds have received much attention due to their wide range of applications in organic and medicinal chemistry. 1,2,3-Triazoles have been used in pharmaceuticals, agrochemicals, dyes and photographic materials<sup>1</sup>. There are numerous examples in the literature of the biological activity of triazole compounds as anti-HIV agents<sup>2</sup> or antibiotic agents<sup>3</sup> and as selective  $\beta_3$  adrenergic agonist receptors<sup>4</sup>. Several methods have been described for the synthesis of 1,2,3-triazoles, but commonly they are available from the thermally induced Huisgen cycloaddition reaction between azides and alkynes<sup>5</sup>. This cycloaddition reaction usually affords mixtures of 1,4- and 1,5-disubstituted 1,2,3-triazoles<sup>6</sup>. K.B.Sharpless and M.Meldal improved the regioselectivity of the reaction by Cu(I) catalyzed ligation (Click Chemistry) of organic azides and terminal alkynes<sup>7,8</sup>. Exclusive 1,4-regioselectivity, mild reaction conditions, effective catalytic system wide substrate scope and high yields are the main advantages of Sharpless click chemistry. In view of wide range of applications of 1,2,3-triazoles we synthesized novel bis-1,2,3-triazole derivatives. Literature survey reveals that bis triazoles are not much explored.

**EXPERIMENTAL SECTION**

***Instrumentation:***

All melting points were measured on a Polmon digital melting point apparatus (Model No MP-96) and were uncorrected. IR spectra were recorded in KBr on Shimadzu-435 spectrophotometer. The <sup>1</sup>H NMR spectra were recorded at 200 MHz on Varian Gemini Unity

Spectrometer using CDCl<sub>3</sub> and DMSO-d<sub>6</sub> solvent with TMS as internal standard (chemical shifts in  $\delta$  ppm). The mass spectra were recorded on Perkin-Elmer Hitachi RDO-62 instrument.

**General procedure for the synthesis of 2,4 bis propargyloxy acetophenones(2a-b):** Resacetophenones **1a-b** (6g, 10 mmol) dissolved in acetone (40ml), propargylbromide (18.8g, 40mmol) was added and stirred at room temperature for 12 hrs. Acetone decanted and evaporated and then diluted with 200ml of ice cold water and extracted twice with ethyl acetate (2x100ml). The combined organic layer was washed with 200ml of water, 200ml of brine and finally dried over solid sodium sulphate, filtered and concentrated under reduced pressure to get the crude material containing compounds **2a-b** as pale pink solid. The solid, which separated on column chromatography (60-120# silica gel) gave compounds **2a-b** in pure form. Recrystallization from pet.ether gives as white needles (yield 68%). The products were characterized from their analytical and spectral data.

*1-[2,4-bis(prop-2-yn-1-yloxy)phenyl]ethanone(2a):* IR (KBr,  $\nu_{\max}$  cm<sup>-1</sup>): 2160 & 2150 (C≡C), 1685 (CO). <sup>1</sup>H NMR (200MHz, CDCl<sub>3</sub>,  $\delta$ , ppm): 2.77 (d, 1H,  $J=2.4$  Hz, C≡C-H), 2.80 (d, 1H,  $J=2.4$  Hz, C≡C-H), 4.79 (d, 2H,  $J=2.4$  Hz, OCH<sub>2</sub>), 4.81 (d, 2H,  $J=2.4$  Hz, OCH<sub>2</sub>), 7.00 (d, 1H,  $J=8.0$  Hz, aromatic), 7.50 (s, 1H, aromatic), 7.90 (d, 1H,  $J=8.0$  Hz, aromatic). MS:  $m/z$  228[M]<sup>+</sup>.

*1-[5-chloro-2,4-bis(prop-2-yn-1-yloxy)phenyl]ethanone (2b):* IR (KBr,  $\nu_{\max}$  cm<sup>-1</sup>): 2165 & 2152 (C≡C), 1690 (CO). <sup>1</sup>H NMR (200MHz, CDCl<sub>3</sub>,  $\delta$ , ppm): 2.6 (s, 3H, COCH<sub>3</sub>), 2.78 (d, 1H,  $J=2.4$  Hz, C≡C-H), 2.81 (d, 1H,  $J=2.4$  Hz, C≡C-H), 4.80 (d, 2H,  $J=2.4$  Hz, OCH<sub>2</sub>), 4.82 (d, 2H,  $J=2.4$  Hz, OCH<sub>2</sub>), 8.00 (s, 1H, aromatic), 7.5 (s, 1H, aromatic). MS:  $m/z$  262[M]<sup>+</sup>.

**General procedure for the Synthesis of 1,4 disubstituted bis 1,2,3 triazoles (3a-j) :** Compounds **2a-b** (10 mmol) and alkyl azides (2.66 mL, 20 mmol) were suspended in a 1:1 mixture of water and t-butyl alcohol (40 mL). Sodium ascorbate (600  $\mu$ L of freshly prepared 1M solution in water) was added, followed by copper (II) sulfate pentahydrate ( 0.06 mmol, in 200  $\mu$ L of water). The heterogeneous mixture was stirred vigorously overnight and TLC analysis indicated complete consumption of the reactants. The reaction mixture was diluted with water (100 mL), cooled in ice, and the solid was collected by filtration. After washing the precipitate with cold water (2 x 25 mL), it was dried under vacuum to afford (**3a-j**).

*1-(2,4-bis((1-benzyl-1H-1,2,3-triazol-4-yl)methoxy)phenyl)ethanone (3a):* m.p. 130-132°C, IR (KBr,  $\nu_{\max}$  cm<sup>-1</sup>): 1685 (CO). <sup>1</sup>H NMR (200MHz, DMSO-d<sub>6</sub>,  $\delta$ , ppm): 2.30 (s, 3H, COCH<sub>3</sub>), 5.10 (s, 2H, benzyl), 5.20 (s, 2H, benzyl), 5.30 (s, 2H, OCH<sub>2</sub>), 5.70 (s, 2H, OCH<sub>2</sub>), 6.80-7.30(m, 11H, aromatic), 7.40 (d,  $J=8.0$  Hz, aromatic), 7.70 (s, 1H, triazole), 7.80 (d,  $J=8.0$  Hz, aromatic), 7.85 (s, 1H, triazole). MS:  $m/z$  494[M]<sup>+</sup>. Analysis: calculated for C<sub>28</sub>H<sub>26</sub>N<sub>6</sub>O<sub>3</sub>: C, 67.94 ; H, 5.25; N, 16.98. Found: C, 67.78; H, 5.18; N, 16.82.

*1-(2,4-bis((1-benzyl-1H-1,2,3-triazol-4-yl)methoxy)-5-chlorophenyl)ethanone(3b):* m.p. 139-142°C, IR (KBr,  $\nu_{\max}$  cm<sup>-1</sup>): 1690 (CO). <sup>1</sup>H NMR (200MHz, DMSO-d<sub>6</sub>,  $\delta$ , ppm): 2.50 (s, 3H, COCH<sub>3</sub>), 5.25 (s, 2H, benzylic), 5.35 (s, 2H, OCH<sub>2</sub>), 5.35(s, 2H, benzylic), 5.70 (s, 2H, OCH<sub>2</sub>), 7.40 (m, 10H, aromatic), 7.50 (s, 1H, aromatic), 7.80 (s, 1H, aromatic), 7.90-8.00 (2s, 2H, triazole protons). MS:  $m/z$  528[M]<sup>+</sup>, 530[M+2]. Analysis: calculated for C<sub>28</sub>H<sub>25</sub>ClN<sub>6</sub>O<sub>3</sub>: C, 63.52 ; H, 4.72; N, 15.86. Found: C, 63.34; H, 4.56; N, 15.60.

*1-(2,4-bis((1-butyl-1H-1,2,3-triazol-4-yl)methoxy)phenyl)ethanone (3c)*: m.p. 128-131°C, IR (KBr,  $\nu_{\text{max}}$   $\text{cm}^{-1}$ ): 1685 (CO).  $^1\text{H}$  NMR (200MHz, DMSO- $d_6$ ,  $\delta$ , ppm): 0.95 (2t, merged, 6H), 1.50 (m, 4H,  $(\text{CH}_2)_2$ ), 1.80(m, 4H,  $(\text{CH}_2)_2$ ), 2.30 (s, 3H,  $\text{COCH}_3$ ), 4.20 (2t, 4H,  $-\text{N}(\text{CH}_2)_2$ ), 6.80 (d, 1H,  $J=8.3$  Hz, aromatic), 7.00 (s, 1H, aromatic), 7.80 (d, 1H,  $J=8.3$ Hz, aromatic), 8.10-8.15( 2s, 2H, triazole). MS:  $m/z$  426 $[\text{M}]^+$ . Analysis: calculated for  $\text{C}_{22}\text{H}_{30}\text{N}_6\text{O}_3$ : C, 61.89 ; H, 7.03; N, 19.69. Found: C, 61.63; H, 6.72; N, 19.47.

*1-(2,4-bis((1-butyl-1H-1,2,3-triazol-4-yl)methoxy)-5-chlorophenyl)ethanone(3d)*: m.p. 135-137°C, IR (KBr,  $\nu_{\text{max}}$   $\text{cm}^{-1}$ ): 1685 (CO).  $^1\text{H}$  NMR (200MHz, DMSO- $d_6$ ,  $\delta$ , ppm): 0.98 (2t, merged, 6H), 1.60 (m, 4H,  $(\text{CH}_2)_2$ ), 1.90(m, 4H,  $(\text{CH}_2)_2$ ), 2.40 (s, 3H,  $\text{COCH}_3$ ), 4.25 (2t, 4H,  $-\text{N}(\text{CH}_2)_2$ ), 7.00 (s, 1H, aromatic), 7.80 (s, 1H, aromatic), 8.10-8.15( 2s, 2H, triazole). MS:  $m/z$  460 $[\text{M}]^+$ , 462 $[\text{M}+2]$ . Analysis: calculated for  $\text{C}_{22}\text{H}_{29}\text{ClN}_6\text{O}_3$ : C, 57.27 ; H, 6.29; N, 18.22. Found: C, 57.05; H, 6.12; N, 18.10.

*2,2'-(4,4'-(4-acetyl-1,3-phenylene)bis(oxy)bis(methylene)bis(1H-1,2,3-triazole-4,1-diyl))bis(1-morpholinoethanone) (3e)*: m.p. 124-126°C, IR (KBr,  $\nu_{\text{max}}$   $\text{cm}^{-1}$ ): 1675 (NC=O), 1685 ( $\text{COCH}_3$ ).  $^1\text{H}$  NMR (200MHz, DMSO- $d_6$ ,  $\delta$ , ppm): 2.30 (s, 3H,  $\text{COCH}_3$ ), 2.92-3.00 (2t, 4H,  $J=5.4$  Hz,  $(\text{COCH}_2)_2$ ), 3.38-3.40 (m, 8H,  $(\text{CH}_2)_4$ ), 3.52 (m, 8H,  $(\text{CH}_2)_4$ ), 4.55-4.60 (2t, 4H,  $J=5.4$  Hz,  $(\text{N}-\text{CH}_2)_2$ ), 5.00 (s, 2H,  $\text{OCH}_2$ ), 5.30 (s, 2H,  $\text{OCH}_2$ ), 7.00 (d, 1H,  $J=8.0$ Hz, aromatic), 7.70 (d, 1H,  $J=8.0$ Hz, aromatic), 7.40 (s, 1H, aromatic) 8.0-8.2(2s, 2H, triazole). MS:  $m/z$  568 $[\text{M}]^+$ . Analysis: calculated for  $\text{C}_{26}\text{H}_{32}\text{N}_8\text{O}_7$ : C, 54.88 ; H, 5.62; N, 19.70. Found: C, 54.63; H, 5.48; N, 19.64.

*2,2'-(4,4'-(4-acetyl-6-chloro-1,3-phenylene)bis(oxy)bis(methylene)bis(1H-1,2,3-triazole-4,1-diyl))bis(1-morpholinoethanone)(3f)*: m.p. 130-131°C, IR (KBr,  $\nu_{\text{max}}$   $\text{cm}^{-1}$ ): 1670(NC=O), 1685 ( $\text{COCH}_3$ ).  $^1\text{H}$  NMR (200MHz, DMSO- $d_6$ ,  $\delta$ , ppm): 2.40 (s, 3H,  $\text{COCH}_3$ ), 2.95- 3.00 (2t, 4H,  $J=5.4$  Hz,  $-(\text{COCH}_2)_2$ ), 3.40-3.43 (m, 8H,  $(\text{CH}_2)_4$ ), 3.55 (m, 8H,  $(\text{CH}_2)_4$ ), 4.58- 4.62 (2t, 4H,  $J=5.4$  Hz,  $(\text{N}-\text{CH}_2)_2$ ), 5.20 (s, 2H,  $\text{OCH}_2$ ), 5.40 (s, 2H,  $\text{OCH}_2$ ), 7.40 (s, 1H, aromatic), 7.80 (s, 1H, aromatic). MS:  $m/z$  602 $[\text{M}]^+$ , 604  $[\text{M}+2]$ . Analysis: calculated for  $\text{C}_{26}\text{H}_{31}\text{ClN}_8\text{O}_7$ : C, 51.73 ; H, 5.14; N, 18.57. Found: C, 51.49; H, 4.93; N, 18.36.

*3,3'-(4,4'-(4-acetyl-1,3-phenylene) bis (oxy) bis (methylene) bis (1H-1,2,3-triazole-4,1-diyl)) bis (1-morpholinopropan-1-one)(3g)*: m.p. 127-130°C, IR (KBr,  $\nu_{\text{max}}$   $\text{cm}^{-1}$ ): 1680(NC=O), 1690 ( $\text{COCH}_3$ ).  $^1\text{H}$  NMR (200MHz, DMSO- $d_6$ ,  $\delta$ , ppm): 2.60 (s, 3H,  $\text{COCH}_3$ ), 3.0-3.3 (2t, 4H,  $J=5.1$ Hz,  $(\text{COCH}_2)_2$ ), 3.32-3.53 (m, 8H,  $(\text{CH}_2)_4$ ), 3.65-3.67 (m, 8H,  $J=4.2$  Hz,  $-(\text{CH}_2)_4$ ), 4.58-4.60(2t, 4H,  $J=5.1$ Hz,  $(\text{NCH}_2)_2$ ), 5.10 (s, 2H,  $\text{OCH}_2$ ), 5.18 (s, 2H,  $\text{OCH}_2$ ), 7.30 (s, 1H, aromatic), 7.50 (d, 1H,  $J=8.0$  Hz, aromatic), 7.80 (d, 1H,  $J=8.0$  Hz, aromatic), 8.00-8.20 (2s, 2H, triazole). MS:  $m/z$  630 $[\text{M}]^+$ . Analysis: calculated for  $\text{C}_{28}\text{H}_{35}\text{ClN}_8\text{O}_7$ : C, 53.24 ; H, 5.54; N, 17.74. Found: C, 53.09; H, 5.30; N, 17.58.

*3,3'-(4,4'-(4-acetyl-6-chloro-1,3-phenylene)bis(oxy)bis(methylene)bis(1H-1,2,3-triazole-4,1-diyl))bis(1-morpholinopropan-1-one)(3h)*: m.p. 135-137°C, IR (KBr,  $\nu_{\text{max}}$   $\text{cm}^{-1}$ ) 1682 (NC=O), 1690 (CO). The  $^1\text{H}$  NMR (200MHz, DMSO- $d_6$ ,  $\delta$ , ppm): 2.70 (s, 3H,  $\text{COCH}_3$ ), 3.0-3.33(2t, 4H,  $J=5.1$ Hz,  $(\text{COCH}_2)_2$ ), (3.40-3.53 (m, 8H,  $\text{N}(\text{CH}_2)_4$ ), 3.67-3.70 (m, 8H,  $-\text{O}(\text{CH}_2)_4$ ), 5.15 (s, 2H,  $\text{OCH}_2$ ), 5.20 (s, 2H,  $\text{OCH}_2$ ), 5.60 (s, 2H,  $\text{N}-\text{CH}_2$ ), 7.40 (s, 1H, aromatic), 7.90 (s, 1H, aromatic),

8.20-8.40 (2s, 2H, triazole). MS:  $m/z$  596[M]<sup>+</sup>, 598 [M+2]. Analysis: calculated for C<sub>28</sub>H<sub>36</sub>N<sub>8</sub>O<sub>7</sub>: C, 56.31 ; H, 6.03; N, 18.77. Found: C, 56.15; H, 5.89; N, 18.62.

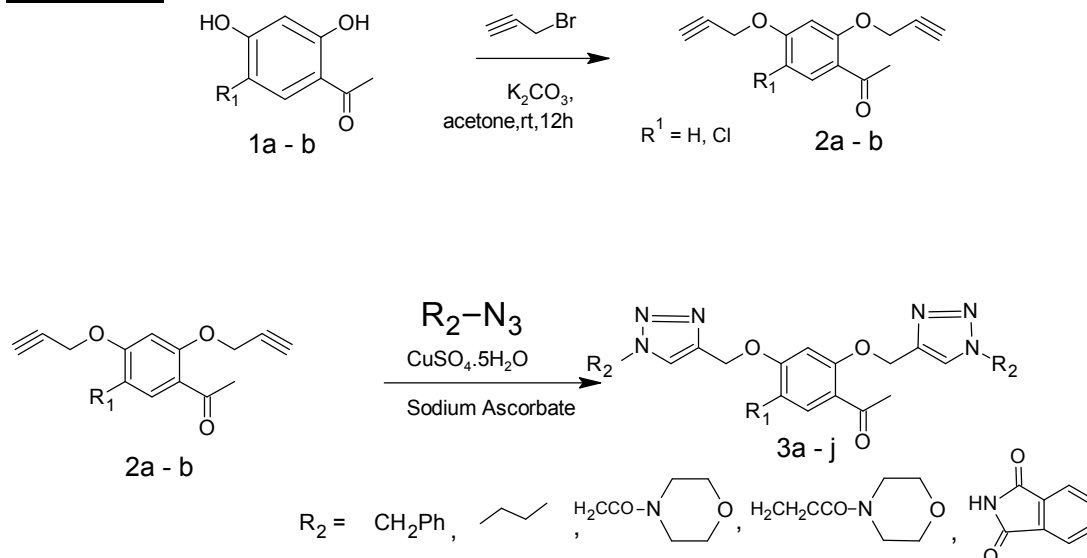
2,2'-(4,4'-(4,4'-(4-acetyl-1,3-phenylene)bis(oxy)bis(methylene)bis(1H-1,2,3-triazole-4,1-diyl))bis(butane-4,1-diyl))diisoindoline-1,3-dione(3i): m.p. 149-151°C, IR (KBr,  $\nu_{\max}$  cm<sup>-1</sup>) 1690 (CO), 1730(CO, phthaloyl). <sup>1</sup>H NMR (200MHz, DMSO-d<sub>6</sub>,  $\delta$ , ppm): 1.80-2.10(m, 8H, (CH<sub>2</sub>)<sub>4</sub>), 2.50 (s, 3H, COCH<sub>3</sub>), 3.80-3.90 (2t, 4H, -N-CH<sub>2</sub>, phthaloyl), 4.25-4.27 (2t, 4H, -N-CH<sub>2</sub>), 5.10-5.20 (2s, 4H, OCH<sub>2</sub>), 6.90 (d, 1H,  $J=8.0$  Hz, aromatic), 7.00-7.40 (m, 10H, aromatic), 7.70 (d, 1H,  $J=8.0$  Hz, aromatic), 7.90-8.00 (2s, 2H, triazole). MS:  $m/z$  716[M]<sup>+</sup>. Analysis: calculated for C<sub>38</sub>H<sub>36</sub>N<sub>8</sub>O<sub>7</sub>: C, 63.62 ; H, 5.02; N, 15.62. Found: C, 63.45; H, 4.85; N, 15.48.

2,2'-(4,4'-(4,4'-(4-acetyl-6-chloro-1,3-phenylene)bis(oxy)bis(methylene)bis(1H-1,2,3-triazole-4,1-diyl))bis(butane-4,1-diyl))diisoindoline-1,3-dione(3j): m.p. 154-156°C, IR (KBr,  $\nu_{\max}$  cm<sup>-1</sup>) 1695 (CO), 1750(CO, phthaloyl). <sup>1</sup>H NMR (200MHz, DMSO-d<sub>6</sub>,  $\delta$ , ppm): 1.80-2.00 (m, 8H, (CH<sub>2</sub>)<sub>4</sub>), 2.40 (s, 3H, COCH<sub>3</sub>), 3.70-3.80 (2t, 4H, -N-CH<sub>2</sub>, phthaloyl), 4.20-4.25 (2t, 4H, -N-CH<sub>2</sub>), 5.00-5.10 (2s, 4H, OCH<sub>2</sub>), 7.00-7.40 (m, 10H, aromatic), 7.70 (s, 1H, aromatic), 7.90-8.10 (2s, 2H, triazole). MS:  $m/z$  750[M]<sup>+</sup>, 752[M+2]. Analysis: calculated for C<sub>38</sub>H<sub>35</sub>ClN<sub>8</sub>O<sub>7</sub>: C, 60.70 ; H, 4.65; N, 14.90. Found: C, 60.56; H, 4.54; N, 14.78.

## RESULTS AND DISCUSSION:

The aim of the present work was to develop simple and efficient procedure for the synthesis of bis 1,2,3-triazoles. The compounds resacetophenones **1a-b** synthesized according to the literature procedure. The key intermediate **2a-b** obtained by treating **1a-b** with propargyl bromide in acetone-K<sub>2</sub>CO<sub>3</sub> medium. The structures of **2a-b** are characterized by spectral analysis. Compound **2a** in its IR showed peaks at 2160 and 2150cm<sup>-1</sup>(C≡C) and 1685 (CO) in the <sup>1</sup>H NMR of **2a** the characteristic protons appeared at  $\delta$  2.50 (s, COCH<sub>3</sub>),  $\delta$  2.70 (d,  $J=2.4$ Hz, C≡CH),  $\delta$  2.80 (d,  $J=2.4$ Hz, C≡CH),  $\delta$  4.79 (d,  $J=2.4$ Hz, OCH<sub>2</sub>),  $\delta$  4.81 (d,  $J=2.4$  Hz, OCH<sub>2</sub>),  $\delta$  7.00 (d,  $J=8.0$  Hz, aromatic),  $\delta$  7.50 (s, aromatic),  $\delta$  7.90 (d,  $J=8.0$ Hz, aromatic), The azides are prepared by the reaction of alkyl halides with sodium azide in water acetone medium (1:4). The compound **2a** on reaction with benzyl azide in presence of CUSO<sub>4</sub>.5H<sub>2</sub>O and sodium ascorbate in water and t-butanol affords exclusively 1,4-regioisomer 1-(2,4-bis((1-benzyl-1H-1,2,3-triazol-4-yl)methoxy)phenyl)ethanone **3a**, similarly **3b-j** are synthesized under similar conditions. The compounds **3a-j** characterized by spectral data. In the IR of **3a** peak appeared at 1685cm<sup>-1</sup>(CO). In the <sup>1</sup>H NMR the proton of the newly formed triazole rings appeared at  $\delta$  7.56 and 7.85 the benzyl protons as singlets at  $\delta$  5.20 and  $\delta$  5.60 and the OCH<sub>2</sub> protons as singlets at  $\delta$  5.30 and  $\delta$  5.70. The aromatic protons  $\delta$  6.80-7.30 (m, aromatic),  $\delta$  7.40 (d,  $J=8.0$  Hz, aromatic),  $\delta$  7.80 (d,  $J=8.0$  Hz, aromatic),

## SCHEME :



**CONCLUSION:** In conclusion we have developed a facile and convenient regioselective synthesis of a series of new 1,4-disubstituted bis 1,2,3-triazoles from bis propargyloxy acetophenones and organic azides catalysed by copper (II) sulfate pentahydrate in presence of sodium ascorbate by the conventional click chemistry.

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