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3-Aryl and 5-Aryl-4-methoxy-6-methyl-2*H*-pyran-2-ones by Suzuki Cross-Coupling Reactions of 3- and 5-Halogeno-4-methoxy-6-methyl-2*H*-pyran-2-ones

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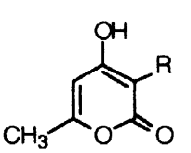
Abstract:

3-Arylpyrones and 5-arylpyrones featuring the 4-methoxy-2*H*-pyran-2-one moiety are obtained by Suzuki cross-coupling of the corresponding 3-iodo and 5-bromo derivatives with arylboronic acids.

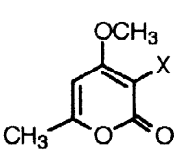
Keywords: Palladium; Catalysis; Pyrones; Suzuki reactions

Introduction

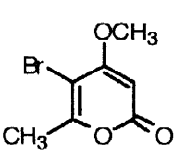
The 4-hydroxy-2*H*-pyran-2-one moiety is a key structure in a recently discovered new type of non-peptidic HIV protease inhibitors [1,2]. Therefore, extension of synthetic methodologies aimed to functionalize the commercially available dehydroacetic acid, **1a**, and triacetic acid lactone, **1b**, is required. The chemistry of **1a,b** and related pyrones has been reviewed [3,4]. Direct arylation of the pyrone ring at the manipulable C-3 and C-5 positions had not been reported till very recently when the palladium-catalyzed cross-coupling of organozinc derivative **2** (X = ZnCl) with iodobenzene was published [5]. Related Pd(0)-catalyzed reactions are the arylation at C-4 of 4-bromo-6-methyl-2*H*-pyran-2-one [5], the cross-couplings on 5-bromo-2*H*-pyran-2-one [6] and on 4-substituted coumarins [7, 8], and the allylation of **1b** at C-3 [9, 10]. Palladium-mediated cross-coupling reactions with 5-halo 2,3-dihydropyran-4-ones [11], 5-halo 2,3-dihydro-4-pyridones [12], and 3-bromochromones [13] have also been reported. Since a vast array of brominated 4-hydroxy- and 4-methoxy-6-methyl-2*H*-pyran-2-ones is available [3, 14], we decided to study the Suzuki-type cross-coupling [15-19] of pyrones **2b,c** and **3** with commercial boronic acids.



1a: R = $COCH_3$
1b: R = H



2a: X = H
2b: X = Br
2c: X = I



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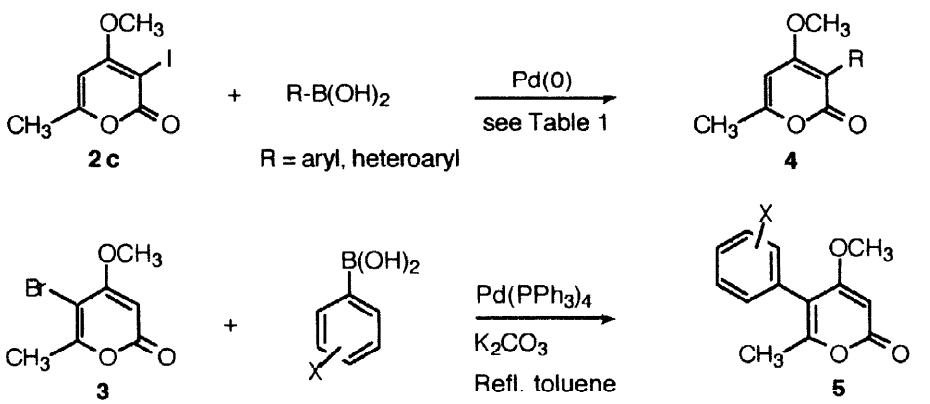
We want to report here our results on the arylation of the C-3 and C-5 positions of these pyrones **2b-c** and **3** by Suzuki cross-couplings.

Results

Initial experiments with bromopyrone **2b** [14] failed. Therefore we prepared 3-iodo-4-methoxy-6-methyl-2*H*-pyran-2-one, **2c**, by reaction of **2a** with *N*-iodosuccinimide in acetonitrile according to a general procedure [20]. After some optimization we found that reactions of **2c** with arylboronic acids were best performed in refluxing toluene with $Pd(dba)_2$ as palladium source, without added phosphine ligands (see Scheme 1 and Table 1). In some cases pyrone **2a** and biaryls derived from the corresponding arylboronic acids were also formed as by-products. Contrarily, some heteroarylboronic acids did not gave the coupling products under these conditions. After some experimentation modest yields were obtained with 3-thiopheneboronic acid and 2-benzo[b]furaneboronic acid using $Pd(PPh_3)_4$ in refluxing DME, longer reaction times being required for the reaction to complete. In the last case (entry 9 in Table 1) an unexpected by-product (8%) was also isolated which showed spectroscopic data according to the structure 3-(2-thienyl)-4-methoxy-6-methyl-2*H*-pyran-2-one.



Scheme 1

Table 1
3-Arylpyrones **4** by Suzuki cross-coupling of **2c** with arylboronic acids.

Entry	R	Conditions[a]	time	**4** (%)[b]	**4** mp (°C)
1	4-MeO-C_6H_4-	A	33 h	**4a** (66)	169-171
2	4-Me-C_6H_4-	A	19 h	**4b** (87)	143-145
3	2-Me-C_6H_4-	A	29 h	**4c** (50)	124-126
4	C_6H_5-	A	86 h	**4d** (70)	156-147
5	4-Cl-C_6H_4-	A	23 h	**4e** (74)	153-154
6	4-CF_3-C_6H_4-	A	74 h	**4f** (62)	164-166
7	4-NO_2-C_6H_4-	A	68 h	**4g** (38)	170-176
8	3-thienyl	B	6 d	**4h** (45)	113-115
9	2-benzo[b]furyl	C	6 d	**4i** (18)[c]	143-145

[a]A: 5% Molar $Pd(dba)_2$ and refluxing toluene; B: 2% molar $Pd(PPh_3)_4$ and refluxing DME; C: 5% molar $Pd(PPh_3)_4$, refluxing DME. [b]Yields of pure isolated products. [c] See text and experimental for by-product.

Initial experiments targeted to arylate C-5 were also carried out with bromopyrone **3** [21] and the results are summarized in Scheme 1 and Table 2. In this case $Pd(PPh_3)_4$ was superior as palladium source.

Table 2
5-Arylpyrones **5** by Suzuki cross-coupling of **3** with arylboronic acids.[a]

Entry	X	time (d)	**5** (%)[b]	**5** mp (°C)
1	4-MeO	11	**5a** (33)	120-122
2	H	2	**5d** (64)	92-94
3	4-Cl	2	**5e** (41)	139-140
4	4-CF_3	11	**5f** (34)	146-147

[a] 5% molar $Pd(PPh_3)_4$ and refluxing toluene in all experiments. [b] Yields of pure isolated products.

In summary, Pd(0)-catalyzed Suzuki cross-coupling is an efficient method to prepare 3- and 5-aryl-4-methoxy-6-methyl-2*H*-pyran-2-ones **4** and **5** from easily available halogenoderivatives of triacetic acid lactone methyl ether.

Experimental

Compound **3** was prepared as described [21]. Mass spectra were registered under electron impact at 70 eV. ^1H NMR (^{13}C NMR) spectra were registered at 250 MHz (62.5 MHz). Tetramethylsilane was used as internal standard. Chemical shifts are given in δ units. Arylboronic acids were purchased from Aldrich or Lancaster. Heteroarylboronic acids were from Lancaster.

3-Iodo-4-methoxy-6-methyl-2*H*-pyran-2-one, 2c.
N-Iodosuccinimide (8.437 g, 37.5 mmole) was added under nitrogen atmosphere to a magnetically stirred solution of **2a** (3.503 g, 25 mmole) in acetonitrile (100 mL). The stirred mixture was maintained for 10 h at room temperature, then the solvent was evaporated and the resulting orange solid was redissolved in dichloromethane. The organic solution was washed with an aqueous solution of sodium tiosulfate (3 x 100 mL), the organic layer was dried with anhydrous sodium sulfate and the solvent was evaporated to yield **2c** as a yellowish solid (5.952 g, 90%); mp 144-146 °C; IR (KBr) 1698 cm^{-1}; ^1H NMR ($CDCl_3$): 2.30 (s, 3H), 3.98 (s, 3H), 5.98 (s, 1H); ^{13}C NMR ($CDCl_3$): 20.0, 57.4, 62.0, 94.7, 161.6, 164.1, 170.4; MS (*m/z*, %): 266 (M^+, 100), 238 (64), 195 (35), 43 (51). Anal.: Calcd. for $C_7H_7IO_3$: C, 31.60; H, 2.65. Found: C, 31.67; H, 2.61.

4-Methoxy-3-(4-methoxyphenyl)-6-methyl-2*H*-pyran-2-one, 4a (General Method).
A mixture of iodopyrone **2c** (0.133 g, 0.50 mmole), 4-methoxyphenylboronic acid (0.091 g, 0.60 mmole), potassium carbonate (0.175 g, 1.24 mmole), $Pd(dba)_2$ (0.013 g, 0.025 mmol), and degassed anhydrous toluene (5 mL) was refluxed for 33 h (GC monitoring) under magnetic stirring and argon atmosphere. Then the mixture was partitioned with diluted aqueous potassium carbonate (2 x 10 mL) and the aqueous phase was extracted with dichloromethane (3 x 10 mL). The combined organic phase (toluene plus dichloromethane) was dried and evaporated to give a residue (0.146 g) which was chromatographed through a column of silica gel (230-400 mesh) with hexanes-ethyl acetate (8:2) to afford **4a** (0.081 g,

66%); mp 169-171 °C (dichloromethane-pentane); IR (KBr): 1683 cm^{-1}; ^1H NMR ($CDCl_3$): 2.32 (s, 3H), 3.82 (s, 3H), 3.83 (s, 3H), 6.13 (s, 1H), 6.92 (d, J = 8.8 Hz, 2H), 7.37 (d, J = 8.8 Hz, 2H); ^{13}C NMR ($CDCl_3$): 20.5, 55.1, 56.5, 95.2, 95.4, 113.4, 123.5, 131.5, 131.7, 158.8, 162.3, 165.6; MS (*m/z*, %): 246 (M^+, 84), 218 (34), 204 (15), 203 (100), 175 (32), 135 (79), 132.1 (15), 89 (15), 43 (48). Anal.: Calcd. for $C_{14}H_{14}O_4$: C, 68.28; H, 5.73. Found: C, 68.32; H, 5.32.

4-Methoxy-6-Methyl-3-(4-methylphenyl)-2*H*-pyran-2-one, 4b.

It was obtained in 87% yield from **2c** and 4-methylphenylboronic acid as for **4a**. Mp 143-145 °C (ethyl acetate-pentane); IR (KBr): 1687 cm^{-1}; ^1H NMR ($CDCl_3$): 2.29 (s, 3H), 2.34 (s, 3H), 3.79 (s, 3H), 6.14 (s, 1H), 7.18 (d, J = 8.0 Hz, 2H), 7.30 (d, J = 8.0 Hz, 2H); ^{13}C NMR ($CDCl_3$): 20.3, 21.2, 56.4, 95.3, 105.0, 128.5, 130.2, 137.0, 162.3, 164.3, 165.8; MS (*m/z*, %): 230 (M^+, 100), 202 (50), 187 (57), 159 (15), 119 (49), 115 (17), 43 (13).

4-Methoxy-6-methyl-2-(2-methylphenyl)-2*H*-pyran-2-one, 4c.

It was obtained in 50% yield from **2c** and 2-methylphenylboronic acid as for **4a** (hexanes-ethyl acetate 7:3 as eluent). Mp 124-126 °C (ethyl acetate-pentane); IR (KBr): 1691 cm^{-1}; ^1H NMR ($CDCl_3$): 2.19 (s, 3H), 2.35 (s, 3H), 3.80 (s, 3H), 6.14 (s, 1H), 7.15-7.26 (m, 4H); ^{13}C NMR ($CDCl_3$): 19.6, 20.4, 56.5, 95.2, 104.9, 125.5, 128.0, 130.0, 130.8, 131.0, 137.6, 163.0, 163.8, 166.3; MS (*m/z*, %): 230 (M^+, 100), 199 (36), 188 (97), 159 (64), 128 (40), 115 (36), 114 (15), 43 (46). Anal.: Calcd. for $C_{14}H_{14}O_3$: C, 73.03; H, 6.13. Found: C, 72.78; H, 5.71.

4-Methoxy-6-methyl-3-phenyl-2*H*-pyran-2-one, 4d.

It was obtained in 70% yield from **2c** and phenylboronic acid as for **4a**. Mp 156-157 °C (dichloromethane-pentane); IR (KBr): 1700 cm^{-1}; ^1H NMR ($CDCl_3$): 2.33 (s, 3H), 3.83 (s, 3H), 6.14 (s, 1H), 7.27-7.45 (m, 5H); ^{13}C NMR ($CDCl_3$): 20.4, 56.5, 95.3, 105.1, 127.3, 127.8, 130.4, 130.7, 162.7, 164.2, 165.9; MS (*m/z*, %): 217 (M + 1, 15), 216 (M^+, 100), 188 (66), 173 (62), 145 (19), 105 (76), 102 (19), 77 (13), 43 (26). Anal.: Calcd. for $C_{13}H_{12}O_3$: C, 72.21; H, 5.59. Found: C, 71.80; H, 5.51.

3-(4-Chlorophenyl)-4-methoxy-6-methyl-2*H*-pyran-2-one, 4e.

It was obtained in 74% yield from **2c** and 4-chlorophenylboronic acid as for **4a**. Mp 153-154 °C (dichloromethane-pentane); IR (KBr) 1705 cm^{-1}; ^1H NMR ($CDCl_3$) d 2.32 (s, 3H), 3.83 (s, 3H), 6.12 (s, 1H), 7.29-7.40 (m, 4H); ^{13}C NMR ($CDCl_3$) d 20.4, 56.6, 95.2, 103.9, 128.0, 129.8, 131.8, 133.0, 163.1, 163.9, 166.1; MS (*m/z*) 252 (34), 250 (M^+, 100), 224 (28), 222 (85), 209 (28), 207 (82), 179 (26), 141 (33), 139 (97), 136 (25), 75 (16), 43 (75). Anal.: Calcd. for $C_{13}H_{11}ClO_3$: C, 62.29; H, 4.42. Found: C, 62.19; H, 4.58.

3-(4-Trifluoromethylphenyl)-4-methoxy-6-methyl-2*H*-pyran-2-one, 4f.

It was obtained in 62% yield from **2c** and 4-trifluoromethylphenylboronic acid as for **4a**. Mp 164-166 °C (dichloromethane-pentane); IR (KBr): 1690 cm^{-1}; ^1H NMR ($CDCl_3$): 2.35 (s, 3H), 3.87 (s, 3H), 6.17 (s, 1H), 7.56 (d, J ca 8.0 Hz, 2H), 7.63 (d, J ca 8.0 Hz, 2H); ^{13}C NMR ($CDCl_3$): 20.6, 56.6, 95.1, 103.8, 124.2 (q, J = 270 Hz), 124.7, 129.1 (q, J = 33 Hz), 130.9, 135.2, 163.7, 163.8, 166.5; MS (*m/z*, %): 284 (M^+, 54), 256 (58), 241 (35), 213 (17), 173 (100), 145 (15), 43 (62). Anal. Calcd. for $C_{14}H_{11}F_3O_3$: C, 59.17; H, 3.90. Found: C, 59.17; H, 3.79.

4-Methoxy-6-methyl-3-(3-nitrophenyl)-2*H*-pyran-2-one, 4g.

It was obtained in 38% yield from **2c** and 3-nitrophenylboronic acid as for **4a** (hexanes-ethyl acetate 7:3 as eluent). Mp 170-176 °C (ethyl acetate-pentane); IR (KBr): 1707 cm^{-1}; ^1H NMR ($CDCl_3$): 2.37 (s, 3H), 3.90 (s, 3H), 6.20 (s, 1H), 7.54 (br dd, J = 8.0 Hz, J = 8.0 Hz, 1H), 7.83 (ddd, J = 8.0 Hz, J = 1.5 Hz, J = 1.5 Hz, 1H), 8.14 (ddd, J = 8.0 Hz, J = 2.2 Hz, J

= 1.5 Hz, 1H), 8.33 (dd, J = 2.2 Hz, J = 1.5 Hz, 1H); ^{13}C NMR ($CDCl_3$): 20.7, 56.8, 95.1, 102.9, 122.2, 125.8, 128.7, 133.2, 136.8, 147.9, 163.5, 164.1, 166.7; MS (*m/z*, %): 261 (M^+, 100), 233 (92), 218 (32), 150 (47), 144 (15), 43 (39). Anal. Calcd. for $C_{13}H_{11}NO_5$: C, 59.77; H, 4.24; N, 5.36. Found: C, 59.41; H, 4.17; N, 5.32.

4-Methoxy-6-methyl-3-(3-thienyl)-2*H*-pyran-2-one, 4h.

It was obtained in 45% yield from **2c** and 3-thiopheneboronic acid under the conditions specified in Table 1 (entry 8). Mp 113-115 °C (ethyl acetate-hexane); IR (KBr): 1700 cm^{-1}; ^{1}H NMR ($CDCl_3$): 2.33 (s, 3H), 3.94 (s, 3H), 6.13 (s, 1H), 7.28 (dd, J = 5.0 Hz, 3.0 Hz, 1H), 7.55 (d, J = 5.0 Hz, 1H), 7.78 (d, J = 3.0 Hz, 1H); ^{13}C NMR ($CDCl_3$): 20.4, 56.5, 95.1, 123.2, 125.4, 129.2, 130.7, 161.8, 163.4, 165.4; MS (*m/z*, %): 222 (M^+, 100), 194 (35), 179 (88), 151 (30), 111 (80). Anal. Calcd. for $C_{11}H_{10}O_3S$: C, 59.45; H, 4.53; S, 14.42. Found: C, 59.32; H, 4.75; S, 13.25.

3-(2-Benzo[b]furyl)-4-methoxy-6-methyl-2*H*-pyran-2-one, 4i.

It was obtained in 18% yield from **2c** and 2-benzo[b]furaneboronic acid under the conditions specified in Table 1 (entry 9). Mp 143-145 °C (isopropanol); IR (KBr): 1690 cm^{-1}; ^{1}H NMR ($CDCl_3$): 2.34 (s, 3H), 3.99 (s, 3H), 6.17 (s, 1H), 7.19-7.29 (m, 2H), 7.22 (broad s, 1H), 7.50 (broad d, J ca 7.5 Hz, 1H), 7.57 (broad d, J ca 7.5 Hz, 1H); ^{13}C NMR ($CDCl_3$): 20.6, 57.0, 95.2, 108.0, 111.0, 120.8, 122.5, 123.9, 128.5, 148.3, 154.3, 163.6, 167.1; MS (m/z, %): 256 (M^+ , 100), 228 (21), 213 (66), 185 (30), 145 (77), 142 (20), 43 (49). A by-product (8%) was also obtained in this reaction with spectroscopic data according to the structure 3-(2-furyl)-4-methoxy-6-methyl-2*H*-pyran-2-one: IR (KBr): 1696 cm^{-1}; ^{1}H NMR ($CDCl_3$): 2.32 (s, 3H), 3.95 (s, 3H), 6.12 (s, 1H), 6.47 (dd, J = 2.9 Hz, 2.2 Hz, 1H), 6.84 (d, J = 2.2 Hz, 1H), 7.50 (d, J = 2.9 Hz, 1H); ^{13}C NMR ($CDCl_3$): 20.5, 56.8, 95.2, 97.0, 110.7, 111.4, 141.6, 145.8, 162.0, 162.4, 165.6; MS (*m/z*, %): 206 (M^+, 100), 178 (22), 163 (62), 135 (32), 95 (52), 43 (40).

4-Methoxy-5-(4-methoxyphenyl)-6-methyl-2*H*-pyran-2-one, 5a (General Method).

A mixture of bromopyrone **3** (0.219 g, 1.00 mmole), 4-methoxyphenylboronic acid (0.182 g, 1.20 mmole), potassium carbonate (0.353 g, 2.50 mmole), $Pd(PPh_3)_4$ (0.058 g, 0.05 mmole), and degassed anhydrous toluene (10 mL) was refluxed for 11 d (GC monitoring) under magnetic stirring and argon atmosphere. Then the mixture was partitioned with diluted aqueous potassium carbonate (2 x 10 mL) and the aqueous phase was extracted with dichloromethane (3 x 10 mL). The combined organic phase (toluene plus dichloromethane) was dried and evaporated to give a residue (0.359 g) which was chromatographed through a column of silica gel (230-400 mesh) with hexanes-ethyl acetate (9:1) to afford **5a** (0.080 g, 33%); mp 120-122 °C (ethyl acetate-pentane); IR (KBr): 1718 cm^{-1}; ^{1}H NMR ($CDCl_3$): 2.09 (s, 3H), 3.74 (s, 3H), 3.84 (s, 3H), 5.52 (s, 1H), 6.91-6.97 (m, 2H), 7.08-7.13 (m, 2H); ^{13}C NMR ($CDCl_3$): 18.1, 55.2, 56.2, 87.5, 113.8, 123.6, 131.5, 159.2, 159.5, 164.2, 170.2; MS (*m/z*, %): 246 (M^+, 100), 231 (87), 218 (33), 203 (21), 175 (51), 43 (53). Anal. Calcd. for $C_{14}H_{14}O_4$: C, 68.28; H, 5.73. Found: C, 68.47; H, 5.82.

4-Methoxy-6-methyl-5-phenyl-2*H*-pyran-2-one, 5d.

It was obtained in 64% yield from **3** and phenylboronic acid as for **5a**. Mp 92-94 °C (dichloromethane-pentane); IR (KBr): 1722 cm^{-1}; ^{1}H NMR ($CDCl_3$): 2.09 (s, 3H), 3.74 (s, 3H), 5.54 (s, 1H), 7.15-7.20 (m, 2H), 7.34-7.45 (m, 3H); ^{13}C NMR ($CDCl_3$): 18.2, 56.1, 87.6, 114.1, 128.0, 128.3, 130.4, 131.7, 159.5, 164.1, 170.0; MS (*m/z*, 5): 216 (M^+, 100), 188 (78), 145 (30), 115 (16), 102 (16), 43 (49). Anal. Calcd. for $C_{13}H_{12}O_3$: C, 72.21; H, 5.59. Found: C, 71.94; H, 5.48.

5-(4-Chlorophenyl)-4-methoxy-6-methyl-2*H*-pyran-2-one, 5e.
It was obtained in 41% yield from **3** and 4-chlorophenylboronic acid as for **5a**. Mp 139-140 °C (ethyl acetate-pentane); IR (KBr): 1723 cm^{-1}; ^1H NMR ($CDCl_3$): 2.09 (s, 3H), 3.74 (s, 3H), 5.53 (s, 1H), 7.10-7.15 (m, 2H), 7.35-7.40 (m, 2H); ^{13}C NMR ($CDCl_3$): 18.2, 59.3, 87.7, 113.2, 128.7, 131.9, 134.2, 159.7, 163.9, 169.7; MS (*m/z*, %): 252 (30), 250 (M^+, 93), 224 (32), 222 (96), 208 (18), 179 (30), 43 (100). Anal. Calcd. for $C_{13}H_{11}ClO_3$: C, 62.29; H, 4.42. Found: C, 62.43; H, 4.49.

5-(4-Trifluoromethylphenyl)-4-methoxy-6-methyl-2*H*-pyran-2-one, 5f.
It was obtained in 34 % yield from **3** and 4-trifluoromethylphenylboronic acid as for **5a**. Mp 146-147 °C (ethyl acetate-pentane); IR (KBr): 1723 cm^{-1}; ^1H NMR ($CDCl_3$): 2.10 (s, 3H), 3.76 (s, 3H), 5.56 (s, 1H), 7.34 (d, J ca 8.0 Hz, 2H), 7.68 (d, J ca 8.0 Hz, 2H); ^{13}C NMR ($CDCl_3$): 18.2, 56.3, 87.8, 113.1, 124.0 (q, J = 274 Hz), 125.3, 130.0, 131.0, 135.6, 159.8, 163.7, 169.4; MS (*m/z*, %): 284 (M^+, 71), 256 (95), 213 (28), 185 (14), 69 (16), 43 (100). Anal. Calcd. for $C_{14}H_{11}F_3O_3$: C, 59.17; H, 3.90. Found: C, 59.08; H, 3.82.

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