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# Synthesis of novel 7-(heteryl/aryl)chromones via Suzuki coupling reaction

**Abstract:** A series of new 7-heteroaryl and arylchromones **6a–I** were synthesized in moderate to good yields by the Suzuki reaction of the triflate (pseudo halide) **5** and a variety of heteroaryl and aryl boronic acids. The resulting products may be used as precursors for synthesis of potentially relevant compounds. The structures of all synthesized compounds were established based on IR, <sup>1</sup>H NMR, <sup>13</sup>C NMR, and DIPMS.

**Keywords:** heteroaryl/arylboronic acids; heteroaryl/arylchromones; pseudo halides; Suzuki coupling.

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## Introduction

Chromones (4*H*-1-benzopyran-4-ones) are naturally occurring oxygen-containing heterocyclic compounds, which perform important biological functions in nature [1] and are a recognized pharmacophore of a large number of bioactive molecules of either natural or synthetic origin. Their derivatives are antibacterial, anticancer, antioxidant, estrogenic agents [2–4], and have been considered as privileged structures in drug development [5]. The Suzuki coupling reaction is the palladium catalyzed C-C bond formation reaction of organoboron compounds with organic halides or pseudo halides [6]. The Suzuki reaction has recently gained much prominence because it is suitable for large-scale synthesis including the industrial synthesis of pharmaceuticals and fine chemicals [7–9]. The key advantages of Suzuki coupling are mild reaction conditions and commercial availability of a wide variety of heterocyclic and arylboronic acids that are safer than other organometallic reagents [10–14]. The Suzuki coupling process tolerates many functional

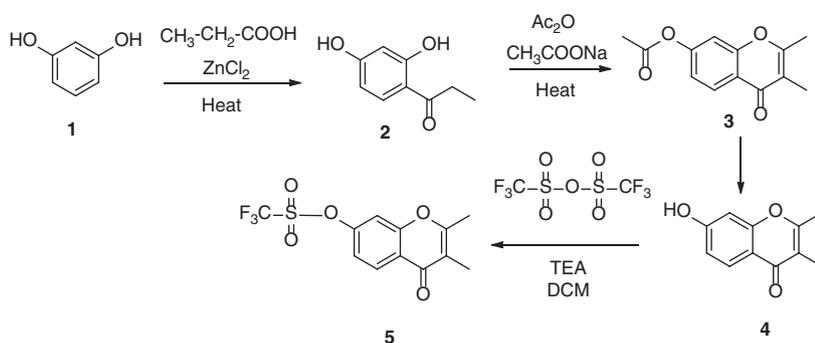
groups present in substrates [15], and it proceeds well in the presence of water. In addition, the inorganic byproduct of the reaction is non-toxic and easily removed from the mixture. This report presents synthesis of 7-heteryl/aryl-2,3-dimethylchromones starting from 7-hydroxy-2,3-dimethylchromones using the Suzuki coupling reaction. To the best of our knowledge, there are no reports in the literature on C-C bond formation at C-7 position of chromones.

## Results and discussion

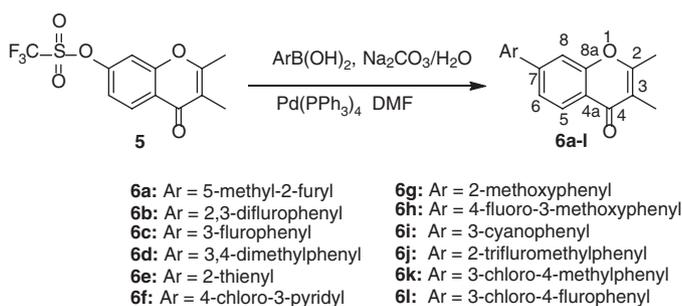
The aim of the present work was to develop a simple and efficient procedure for the preparation of new chromone derivatives bearing substituted heteroaryl and aryl moieties by Suzuki cross coupling reactions. Aryl triflates are good leaving groups that are more reactive than aryl chlorides, bromides, and more stable to moisture and air. The key intermediate chromenyl triflate **5** was prepared by a conventional method, which involves treatment of the corresponding 7-hydroxy-2,3-dimethylchromone **4** with triflic anhydride in the presence of base triethylamine [16, 17]. Compound **4** [18, 19] was prepared from resorcinol according to a procedure in the literature (Scheme 1). The structure of chromenyl triflate **5** was established by spectral analysis. The reaction of the substrate **5** with boronic acids and esters in the presence of Pd(PPh<sub>3</sub>)<sub>4</sub>, sodium carbonate in *N,N*-dimethylformamide under mild conditions afforded the corresponding heteroaryl and arylchromones **6a–I** in moderate to good yields (Scheme 2). Because aryl triflates are sensitive to strong bases, sodium carbonate was used to carry out the Suzuki reaction. The structures of compounds **6a–I** were confirmed by spectral analysis.

## Conclusions

A simple and efficient method of synthesis of heteryl- and aryl-substituted chromones **6a–I** using the Suzuki cross coupling reaction was described.



**Scheme 1** Synthesis of 2,3-dimethyl-4-oxo-4H-chromen-7-yl trifluoromethanesulfonate (5).



**Scheme 2** Synthesis of 7-(aryl/heteryl)-substituted chromones 6a–l.

## Experimental

Chromenyl triflate 5 [16, 17] and 7-hydroxy-2,3-dimethylchromone 4 [18, 19] were prepared by the reported procedures. Melting points were obtained on a Polmon instrument, model MP 96, and were uncorrected. IR spectra were recorded in KBr pellets on a Fourier transform Perkin-Elmer model 337 instrument.  $^1\text{H}$  NMR (400 MHz) and  $^{13}\text{C}$  NMR (100.6 MHz) spectra were recorded in  $\text{CDCl}_3$  solution on a Bruker 400 spectrometer using TMS as an internal standard. Mass spectral data were obtained with an Agilent-6310 ion trap mass spectrometer.

## General procedure for synthesis of 7-(aryl and heteroaryl)-2,3-dimethyl-4-chromones 6a–l

A mixture of chromenyl triflate 5 (0.8 mmol), a boronic acid (1.2 mmol), and aqueous  $\text{Na}_2\text{CO}_3$  (1 mL, 2 M) in DMF was purged with nitrogen with stirring for 30 min, and then treated with 4 mol% of tetrakis(triphenylphosphine)palladium(0). The reaction mixture was stirred at  $60^\circ\text{C}$  for 3–4 h. After completion of the reaction, the mixture was cooled to room temperature, diluted with water (50 mL), and extracted with diethyl ether ( $3 \times 30$  mL). The extract was dried over  $\text{Na}_2\text{SO}_4$  and concentrated under reduced pressure to give crude compounds 6a–l, which were purified by column chromatography on silica gel.

**2,3-Dimethyl-7-(5-methyl-2-furyl)-4H-4-chromone (6a)** Yield 60%; white solid; mp  $123^\circ\text{C}$ ; IR:  $1633.0\text{ cm}^{-1}$  (C=O);  $^1\text{H}$  NMR:  $\delta$  8.14 (d,  $J = 8.3$  Hz, H-5), 7.60 (m, H-6, H-8), 6.71 (d,  $J = 3.0$  Hz, H-3'), 6.10 (d,  $J =$

3.0 Hz, H-4'), 2.40 (s,  $\text{CH}_3$ -2), 2.39 (s,  $\text{CH}_3$ -5'), 2.05 (s,  $\text{CH}_3$ -3);  $^{13}\text{C}$  NMR:  $\delta$  177.6 (C=O), 166.9 (C-2'), 161.8 (C-2), 156.4 (C-8a), 156.2 (C-5'), 142.4 (C-7), 128.4 (C-4'), 124.8 (C-3'), 124.4 (C-4a), 123.5 (C-5), 121.4 (C-6), 116.7 (C-3), 115.1 (C-8), 20.2 ( $5'$ - $\text{CH}_3$ ), 18.7 (2- $\text{CH}_3$ ), 10.2 (3- $\text{CH}_3$ ). HR-ESI-MS. Calcd for  $\text{C}_{16}\text{H}_{14}\text{O}_3$ :  $m/z$  254.0943, found:  $m/z$  254.0938.

**7-(2,3-Difluorophenyl)-2,3-dimethyl-4H-4-chromone (6b)** Yield 70%; mp  $136^\circ\text{C}$ ; IR:  $1638.0\text{ cm}^{-1}$  (C=O);  $^1\text{H}$  NMR:  $\delta$  8.28 (d,  $J = 8.0$  Hz, H-5), 7.61 (d,  $J = 1.2$  Hz, H-8), 7.54 (dd,  $J = 8.0$  Hz,  $J = 1.2$  Hz, H-6), 7.29–7.20 (m, H-3', H-4', H-5'), 2.46 (s,  $\text{CH}_3$ -2), 2.11 (s,  $\text{CH}_3$ -3);  $^{13}\text{C}$  NMR:  $\delta$  177.4 (C=O), 162.2 (C-2), 155.6 (C-8a), 148.2 (C-2'), 142.7 (C-3'), 143.6 (C-5), 136.7 (C-7), 129.6 (C-1'), 127.3 (C-6'), 126.7 (C-5'), 123.3 (C-4a), 121.7 (C-4'), 117.3 (C-6), 117.1 (C-3), 115.4 (C-8), 18.6 (2- $\text{CH}_3$ ), 10.1 (3- $\text{CH}_3$ ). HR-ESI-MS. Calcd for  $\text{C}_{17}\text{H}_{12}\text{F}_2\text{O}_2$ :  $m/z$  286.0805, found:  $m/z$  286.0801.

**7-(3-Fluorophenyl)-2,3-dimethyl-4H-4-chromone (6c)** Yield 58%; mp  $107^\circ\text{C}$ ; IR:  $1630.0\text{ cm}^{-1}$  (C=O);  $^1\text{H}$  NMR:  $\delta$  8.25 (d,  $J = 8.0$  Hz, H-5), 7.60 (d,  $J = 1.2$  Hz, H-8), 7.56–7.49 (m, H-6, H-2'), 7.43–7.37 (m, H-5'), 7.28–7.23 (m, H-4', H-6'), 2.45 (s,  $\text{CH}_3$ -2), 2.10 (s,  $\text{CH}_3$ -3);  $^{13}\text{C}$  NMR:  $\delta$  176.1 (C=O), 161.5 (C-2), 155.5 (C-8a), 148.2 (C-3'), 142.7 (C-1'), 144.8 (C-5), 134.9 (C-7), 129.6 (C-2'), 127.3 (C-6'), 126.7 (C-5'), 123.4 (C-4a), 122.6 (C-4'), 118.1 (C-6), 117.4 (C-3), 115.6 (C-8), 18.6 (2- $\text{CH}_3$ ), 10.1 (3- $\text{CH}_3$ ). HR-ESI-MS. Calcd for  $\text{C}_{17}\text{H}_{12}\text{FO}_2$ :  $m/z$  268.0900, found:  $m/z$  268.0903.

**7-(3,4-Dimethylphenyl)-2,3-dimethyl-4H-4-chromone (6d)** Yield 62%; mp  $89^\circ\text{C}$ ; IR:  $1628.0\text{ cm}^{-1}$  (C=O);  $^1\text{H}$  NMR:  $\delta$  8.21 (d,  $J = 8.0$  Hz, H-5), 7.58 (d,  $J = 1.6$  Hz, H-8), 7.56 (s, H-2'), 7.43–7.38 (m, H-6', H-5', H-4'), 7.24 (d,  $J = 8.0$  Hz, H-6), 2.43 (s,  $\text{CH}_3$ -2), 2.35 (s,  $\text{CH}_3$ -1'), 2.32 (s,  $\text{CH}_3$ -2'), 2.07 (s,  $\text{CH}_3$ -3);  $^{13}\text{C}$  NMR:  $\delta$  177.8 (C=O), 161.8 (C-2), 156.2 (C-8a), 146.1 (C-3'), 137.3 (C-4'), 137.2 (C-7), 136.9 (C-1'), 130.3 (C-2'), 128.5 (C-5'), 126.2 (C-6'), 124.7 (C-4a), 123.5 (C-5), 121.1 (C-6), 116.9 (C-3), 115.2 (C-8),

19.9 (4'-CH<sub>3</sub>), 19.5 (3'-CH<sub>3</sub>), 18.6 (2-CH<sub>3</sub>), 10.1 (3-CH<sub>3</sub>). HR-ESI-MS. Calcd for C<sub>19</sub>H<sub>18</sub>O<sub>2</sub><sup>+</sup>: m/z 278.1307, found: m/z 278.1301.

**2,3-Dimethyl-7-(2-thienyl)-4H-4-chromone (6e)** Yield 56%; mp 112°C; IR: 1635.0 cm<sup>-1</sup> (C=O); <sup>1</sup>H NMR: δ 8.15 (d, *J* = 8.0 Hz, H-5), 7.58–7.55 (m, H-6, H-8), 7.43 (d, *J* = 3.0 Hz, H-3'), 7.37 (dd, *J* = 3.0 Hz, *J* = 3.6 Hz, H-4'), 7.11 (d, *J* = 3.6 Hz, H-5'), 2.40 (s, CH<sub>3</sub>-2), 2.05 (s, CH<sub>3</sub>-3); <sup>13</sup>C NMR: δ 177.4 (C=O), 161.9 (C-2), 156.2 (C-8a), 142.4 (C-7), 138.9 (C-2'), 128.4 (C-4'), 126.7 (C-5'), 126.5 (C-5'), 124.9 (C-3'), 124.7 (C-4a), 123.5 (C-5), 121.1 (C-6), 116.9 (C-3), 115.2 (C-8), 18.6 (2-CH<sub>3</sub>), 10.1 (3-CH<sub>3</sub>). HR-ESI-MS. Calcd for C<sub>15</sub>H<sub>12</sub>O<sub>2</sub>S<sup>+</sup>: m/z 256.0558, found: m/z 256.0547.

**2,3-Dimethyl-7-(4-chloro-3-pyridyl)-4H-4-chromone (6f)** Yield 64%; mp 145°C; IR: 1632.0 cm<sup>-1</sup> (C=O); <sup>1</sup>H NMR: δ 8.66 (s, H-6'), 8.28 (d, *J* = 8.4 Hz, H-5), 7.90 (dd, *J* = 8.4 Hz, *J* = 2.8 Hz, H-4'), 7.56 (d, *J* = 1.6 Hz, H-8), 7.52 (dd, *J* = 8.4 Hz, *J* = 1.6 Hz, H-6), 7.45 (d, *J* = 8.4 Hz, H-3'), 2.44 (s, CH<sub>3</sub>-2), 2.08 (s, CH<sub>3</sub>-3); <sup>13</sup>C NMR: δ 177.4 (C=O), 162.2 (C-2), 156.1 (C-8a), 151.5 (C-2') 148.1 (C-6'), 141.1 (C-5'), 137.3 (C-7), 134.0 (C-4'), 127.0 (C-6), 124.5 (C-4a), 123.2 (C-5), 122.1 (C-3'), 117.4 (C-3), 115.8 (C-8), 18.6 (2-CH<sub>3</sub>), 10.1 (3-CH<sub>3</sub>). HR-ESI-MS. Calcd for C<sub>16</sub>H<sub>12</sub><sup>35</sup>ClNO<sub>2</sub><sup>+</sup>: m/z 285.0557, found: m/z 285.0548.

**2,3-Dimethyl-7-(2-methoxyphenyl)-4H-4-chromone (6g)** Yield 60%; mp 92°C; IR: 1634.0 cm<sup>-1</sup> (C=O); <sup>1</sup>H NMR: δ 8.19 (dd, *J* = 8.2 Hz, *J* = 0.3 Hz, H-6), 7.57 (d, *J* = 0.3 Hz, H-8), 7.51 (dd, *J* = 8.1 Hz, *J* = 1.7 Hz, H-6'), 7.38 (m, H-4', H-3'), 7.05 (m, H-5', H-5), 3.84 (s, OCH<sub>3</sub>), 2.43 (s, CH<sub>3</sub>-2), 2.08 (s, CH<sub>3</sub>-3). <sup>13</sup>C NMR: δ 177.9 (C=O), 161.9 (C-2), 156.5 (C-2'), 155.7 (C-8a), 143.6 (C-7), 130.8 (C-6'), 129.7 (C-1'), 128.9 (C-4'), 126.2 (C-5), 125.2 (C-4a), 121.1 (C-6), 121.0 (C-5'), 118.2 (C-3), 116.9 (C-8), 111.4 (C-3'), 55.6 (OCH<sub>3</sub>), 18.6 (2-CH<sub>3</sub>), 10.1 (3-CH<sub>3</sub>). HR-ESI-MS. Calcd for C<sub>18</sub>H<sub>16</sub>O<sub>3</sub><sup>+</sup>: m/z 280.1099, found: m/z 280.1088.

**7-(4-Fluoro-3-methoxyphenyl)-2,3-dimethyl-4H-4-chromone (6h)** Yield 71%; mp 118°C; IR: 1625.0 cm<sup>-1</sup> (C=O); <sup>1</sup>H NMR: δ 8.23 (d, *J* = 8.2 Hz, H-6), 7.53 (m, H-8, H-5), 7.19 (m, H-2', H-5', H-6'), 3.98 (s, OCH<sub>3</sub>), 2.44 (s, CH<sub>3</sub>-2), 2.08 (s, CH<sub>3</sub>-3); <sup>13</sup>C NMR: δ 177.1 (C=O), 162.0 (C-2), 156.1 (C-8a), 151.6 (C-3'), 148.0 (C-4'), 145.2 (C-1'), 136.1 (C-7), 126.4 (C-5), 123.5 (C-6), 121.4 (C-6'), 120.0 (C-5'), 117.1 (C-4a), 116.4 (C-2'), 115.5 (C-3), 112.6 (C-8), 56.4 (OCH<sub>3</sub>), 18.6 (2-CH<sub>3</sub>), 10.1 (3-CH<sub>3</sub>). HR-ESI-MS. Calcd for C<sub>18</sub>H<sub>15</sub>FO<sub>3</sub><sup>+</sup>: m/z 298.1005, found: m/z 298.1001.

**7-(3-Cyanophenyl)-2,3-dimethyl-4H-4-chromone (6i)** Yield 76%; mp 96°C; IR: 1635.0 cm<sup>-1</sup> (C=O); 2212.0 cm<sup>-1</sup> (CN); <sup>1</sup>H NMR: δ 8.28 (d, *J* = 8.2 Hz, H-6), 7.93 (d, *J* = 1.4 Hz, H-8), 7.87 (dd, *J* = 7.4 Hz, *J* = 1.3

Hz, H-4'), 7.71 (dd, *J* = 7.4 Hz, *J* = 7.6 Hz, H-5'), 7.58 (m, H-5, H-2', H-6'), 2.45 (s, CH<sub>3</sub>-2), 2.09 (s, CH<sub>3</sub>-3); <sup>13</sup>C NMR: δ 177.5 (C=O), 162.2 (C-2), 156.1 (C-8a), 143.4 (C-7), 140.7 (C-1'), 131.8 (C-4'), 131.6 (C-2'), 130.9 (C-6'), 129.9 (C-5'), 126.9 (C-4a), 123.3 (C-5), 122.1 (C-6), 118.1 (CN), 117.4 (C-3), 116.0 (C-8), 113.3 (C-3'), 18.6 (2-CH<sub>3</sub>), 10.1 (3-CH<sub>3</sub>). HR-ESI-MS. Calcd for C<sub>18</sub>H<sub>13</sub>NO<sub>2</sub><sup>+</sup>: m/z 275.0946, found: m/z 275.0937.

**7-(2-Trifluoromethylphenyl)-2,3-dimethyl-4H-4-chromone (6j)** Yield 70%; mp 128°C; IR: 1630.0 cm<sup>-1</sup> (C=O); <sup>1</sup>H NMR: δ 8.21 (dd, *J* = 8.1 Hz, *J* = 0.2 Hz, H-6'), 7.78 (d, *J* = 7.8 Hz, H-5), 7.90 (dd, *J* = 8.4 Hz, *J* = 2.8 Hz, H-4'), 7.56 (d, *J* = 1.6 Hz, H-8), 7.52 (dd, *J* = 8.4 Hz, *J* = 1.6 Hz, H-6), 7.45 (d, *J* = 8.4 Hz, H-3'), 2.44 (s, CH<sub>3</sub>-2), 2.08 (s, CH<sub>3</sub>-3); <sup>13</sup>C NMR: δ 177.5 (C=O), 162.2 (C-2), 156.1 (C-8a), 143.4 (C-1'), 140.7 (C-7), 134.4 (C-2'), 130.6 (C-5'), 129.7 (C-6'), 127.4 (C-3'), 126.7 (C-4a), 123.3 (C-5), 122.1 (C-6), 118.1 (CF<sub>3</sub>), 117.4 (C-3), 116.2 (C-8), 113.4 (C-3'), 18.4 (2-CH<sub>3</sub>), 10.1 (3-CH<sub>3</sub>). HR-ESI-MS. Calcd for C<sub>18</sub>H<sub>13</sub>F<sub>3</sub>O<sub>2</sub><sup>+</sup>: m/z 318.0869, found: m/z 318.0864.

**7-(3-Chloro-4-methylphenyl)-2,3-dimethyl-4H-4-chromone (6k)** Yield 70%; mp 136°C; IR: 1632.0 cm<sup>-1</sup> (C=O); <sup>1</sup>H NMR: δ 8.22 (dd, *J* = 8.2 Hz, *J* = 1.4 Hz, H-6), 7.46 (d, *J* = 1.4 Hz, H-8), 7.40 (d, *J* = 7.8 Hz, H-5'), 7.33 (d, *J* = 7.8 Hz, H-6'), 7.46 (d, *J* = 8.2 Hz, H-5), 7.15 (s, H-2'), 2.43 (s, CH<sub>3</sub>-2), 2.39 (s, CH<sub>3</sub>), 2.08 (s, CH<sub>3</sub>-3); <sup>13</sup>C NMR: δ 177.1 (C=O), 162.0 (C-2), 155.5 (C-8a), 144.2 (C-1'), 139.8 (C-7), 135.9 (C-4'), 131.9 (C-3'), 130.9 (C-2'), 130.6 (C-5'), 127.9 (C-6'), 126.1 (C-4a), 125.5 (C-5), 121.5 (C-6), 118.4 (C-3), 117.1 (C-8), 20.9 (4'-CH<sub>3</sub>), 18.6 (2-CH<sub>3</sub>), 10.1 (3-CH<sub>3</sub>). HR-ESI-MS. Calcd for C<sub>18</sub>H<sub>15</sub><sup>35</sup>ClO<sub>2</sub><sup>+</sup>: m/z 298.0761, found: m/z 298.0765.

**7-(3-Chloro-4-fluoro-phenyl)-2,3-dimethyl-4H-4-chromone (6l)** Yield 67%; mp 140°C; IR: 1627.0 cm<sup>-1</sup> (C=O); <sup>1</sup>H NMR: δ 8.24 (dd, *J* = 8.2 Hz, *J* = 1.4 Hz, H-6), 7.68 (d, *J* = 1.4 Hz, H-8), 7.52 (m, H-2', H-5', H-6'), 7.25 (d, *J* = 8.2 Hz, H-5), 2.44 (s, CH<sub>3</sub>-2), 2.08 (s, CH<sub>3</sub>-3); <sup>13</sup>C NMR: δ 177.6 (C=O), 162.1 (C-2), 159.6 (C-8a), 157.1 (C-7), 156.1 (C-1'), 143.6 (C-5), 136.7 (C-6'), 129.6 (C-2'), 127.1 (C-3'), 126.7 (C-5'), 123.3 (C-4a), 121.7 (C-4'), 117.3 (C-6), 117.0 (C-4a), 115.6 (C-8), 18.6 (2-CH<sub>3</sub>), 10.1 (3-CH<sub>3</sub>). HR-ESI-MS. Calcd for C<sub>17</sub>H<sub>12</sub>F<sup>35</sup>ClO<sub>2</sub><sup>+</sup>: m/z 302.0510, found: m/z 302.0506.

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