

# Synthesis of new *cis*- and *trans*-3-hydroxy-2-(1-phenyl-3-aryl-4-pyrazolyl) chromanones using a hypervalent iodine oxidation approach

Om Prakash\* and Rajesh Kumar

Department of Chemistry, Kurukshetra University, Kurukshetra 136119, India

E-mail: [dromprakash50@rediffmail.com](mailto:dromprakash50@rediffmail.com)

---

## Abstract

Oxidation of 2'-hydroxychalcone analogues of pyrazole **1** using iodobenzene diacetate in KOH-MeOH leads to the formation of *cis*-3-hydroxy-2-(1-phenyl-3-aryl-4-pyrazolyl) chromanone dimethylacetals **2**. The hydrolysis of the dimethylacetals **2** affords either *trans*-3-hydroxy-2-(1-phenyl-3-aryl-4-pyrazolyl) chromanones **3** and *cis*-3-hydroxy-2-(1-phenyl-3-aryl-4-pyrazolyl) chromanones **4** depending upon the hydrolytic conditions in good yields. Their <sup>1</sup>H NMR, IR and mass spectra confirmed the structures of the compounds **2**, **3** and **4**.

**Keywords:** Hypervalent iodine, oxidation, chromanones, iodobenzene diacetate

---

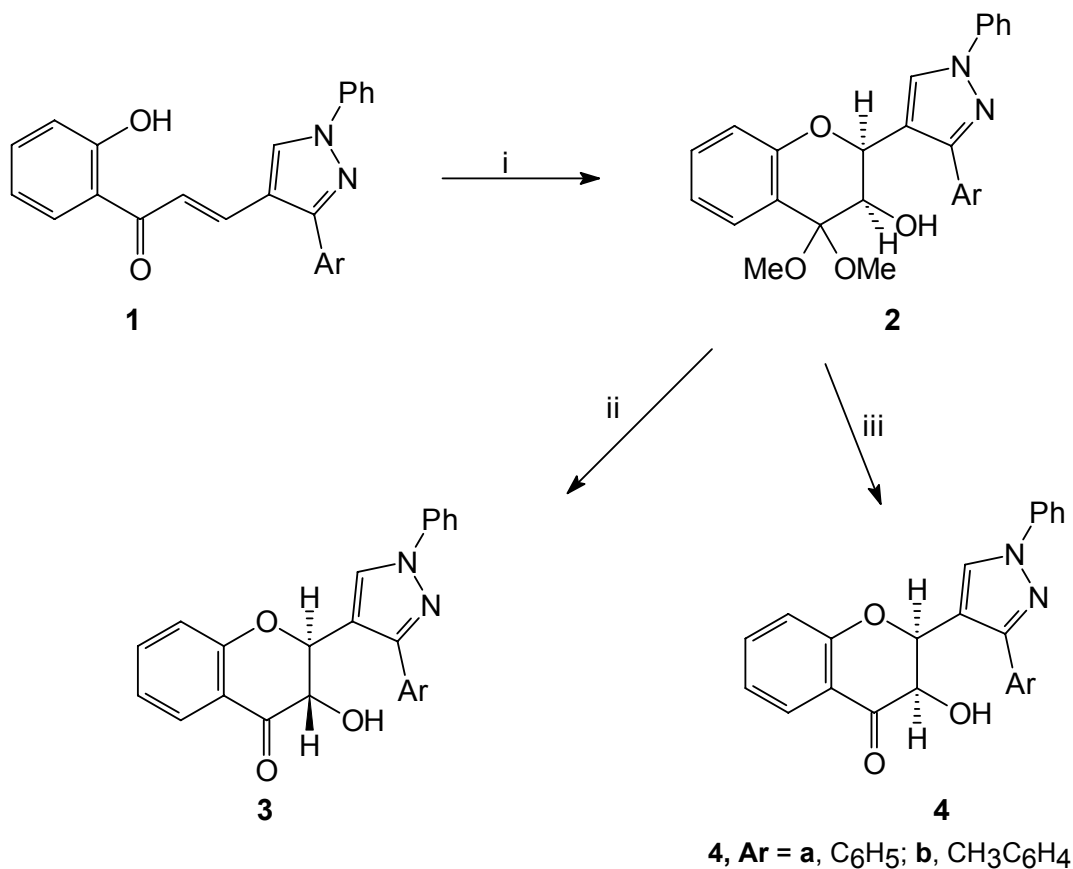
## Introduction

In recent years, considerable attention has been devoted to the use of hypervalent iodine reagents in organic synthesis.<sup>1</sup> Particularly, hypervalent iodine reagents such as iodobenzene diacetate (IBD),<sup>2</sup> iodobis(trifluoro)acetate (IBTA),<sup>3</sup> [hydroxy(tosyloxy)iodo] benzene (HTIB, Koser's Reagent),<sup>4</sup> IBX<sup>5</sup> and Dess-Martin reagent<sup>6</sup> have been used as versatile oxidizing agents. Previous investigations from our laboratory have shown that IBD, HTIB, etc. find interesting applications in flavonoids.<sup>7-14</sup> Among the various such applications, one remarkable use of IBD-KOH/MeOH system<sup>15-17</sup> is the availability of *cis*-3-hydroxyflavanones dimethylacetals, which can be converted into the corresponding *trans*-3-hydroxyflavanones and *cis*-3-hydroxyflavanones depending upon the hydrolytic conditions. *cis*-3-Hydroxyflavanones cannot be synthesized via conventional methods and pyrazole analogues of *cis*- and *trans*-3-hydroxyflavanones of the type **3** and **4** are unknown in literature. Pyrazole<sup>18</sup> and flavanone<sup>19</sup> derivatives are well known for their various biological properties. There has been particular interest in the synthesis of 3-hydroxyflavanone derivatives, having both these moieties i.e. flavanone and pyrazole. Therefore, it was considered worthwhile to undertake the oxidation of 2'-hydroxychalcone

analogues of pyrazole with iodobenzene diacetate (IBD) in KOH-MeOH for synthesizing new *trans*-3-hydroxychromanones **3** and *cis*-3-hydroxychromanones **4** of potential biological interest.

## Results and Discussion

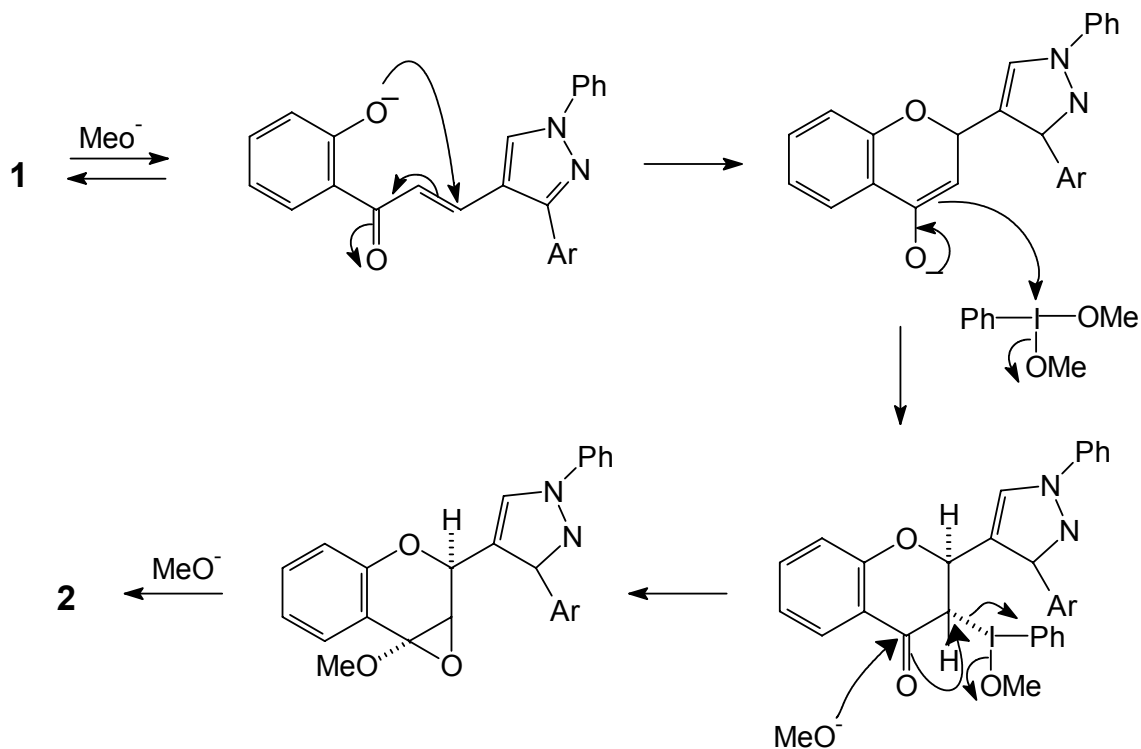
In continuation of these encouraging results, we carried out the reaction of 1-(2-hydroxyphenyl)-3-(1-phenyl-3-aryl-4-pyrazolyl)-2-propen-1-ones **1** with 1.1 equivalents of iodobenzene diacetate (IBD) and 3 equivalents of potassium hydroxide in methanol stirring at 5-10 °C. Usual work-up of the reaction afforded the corresponding dimethylacetals **2** in good yields. The principal objective of this study was to obtain *cis*-3-hydroxychromanones **4**. This was accomplished by the hydrolysis of the dimethylacetals **2** in acetic acid. The use of acetone-conc. HCl instead of acetic acid in this reaction afforded *trans*-3-hydroxychromanones **3**.



Reaction conditions : (i) C<sub>6</sub>H<sub>5</sub>(OAc)<sub>2</sub>, KOH/MeOH, 5-10 °C; (ii) concentrated HCl-acetone, 2h; (iii) 50% AcOH, 50-60 °C, 6h, or 3N HCl/EtOH, 30 min.

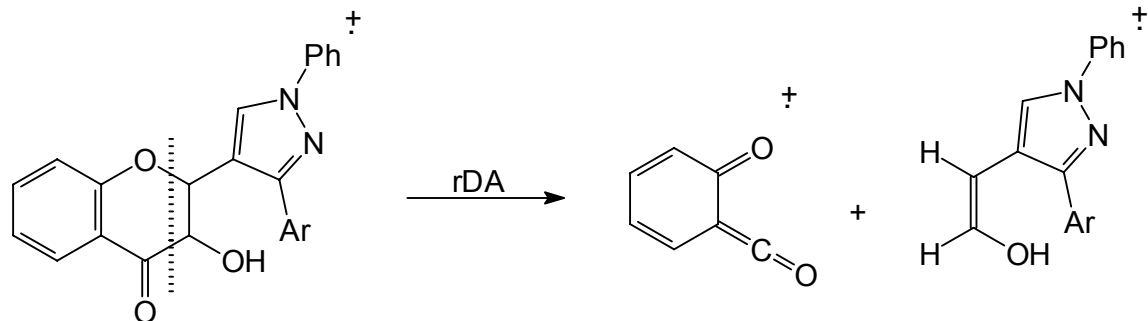
## Scheme 1

The probable mechanism for the transformation of **1** → **2** is analogous to our previous work and is outlined in Scheme 2. This mechanism accounts for the *cis* relationship between C(2)-H and C(3)-H which has been supported by <sup>1</sup>H NMR data.



**Scheme 2**

The structures of the dimethylacetals **2**, *trans*-3-hydroxychromanones **3** and *cis*-3-hydroxychromanones **4** were confirmed by the combined use of <sup>1</sup>H NMR, IR and mass spectra. In <sup>1</sup>H NMR of dimethylacetals **2**, the C(3) proton couple with hydroxy proton split into doublet and C(2) proton has a broad singlet. On shaking with D<sub>2</sub>O, hydroxy proton disappeared and the C(2)- C(3) proton-proton couple together split into doublet of coupling constant 5-6 Hz which agrees with the assigned *cis* stereochemistry. The <sup>1</sup>H NMR spectra of **3** and **4** enabled differentiation between the two. The coupling constant between the C(2) proton centered at 5.35 ppm and the C(3) proton centered at 4.82 ppm was 5.1 Hz for *cis* isomer **3** and for *trans* isomer **4** was 12.6 Hz.



Scheme 3

The mass spectral fragmentation pattern for **3** relative to **4** did not show significant mutual differences. An important fragmentation was the retro-Diels-Alder cleavage, which has been described by Buset and Scheline<sup>20</sup> in some 3-hydroxyflavanones. Similarly the rDA pathway is also followed by compound **2**.

## Conclusions

Finally, it is clear from the results of the present study that the I(III) mediated approach offers an easy and simple method for synthesizing new *trans*- and *cis*-3-hydroxychromanones **3** and **4** as well as their dimethylacetals **2**.

## Experimental Section

**General Procedures.** Melting points were determined in open capillaries with electrical melting point apparatus and are uncorrected. The IR spectra were obtained with a Buck Scientific IR M-500 spectrophotometer. The <sup>1</sup>H NMR spectra were recorded on a Bruker (300 MHz) spectrometer using tetramethylsilane as an internal standard. Elemental analysis (CHN) and mass spectra were performed by RSIC, Central Drug Research Institute, Lucknow, India. Elemental analyses were recorded on Elementar Vario EL III Carlo Erba 1108 analyzer. All the new compounds gave satisfactory analytical results (with in  $\pm 0.4$  of the theoretical values).

1-(2-hydroxyphenyl)-3-(1-phenyl-3-aryl-4-pyrazolyl)-2-propen-1-ones (**1**) were obtained by the condensation of 2'-hydroxyacetophenone with 1-phenyl-3-arylpyrazole-4-carboxaldehydes in KOH-MeOH.<sup>21</sup>

**General procedure for preparation of *cis*-3-hydroxy-2-(1-phenyl-3-aryl-4-pyrazolyl)chromanone dimethylacetals (**2**).** A solution of 1-(2-hydroxyphenyl)-3-(1-phenyl-3-aryl-4-pyrazolyl)-2-propen-1-ones (**1**) (0.01 mol) in methanol (50 ml) was added dropwise to a stirred

solution of potassium hydroxide (0.03 mol) in 30 ml of methanol over a period of 15 min at 5-10 °C. The resulting yellow-brown solution was stirred for 10 min and then was added iodobenzene diacetate (0.011 mol) in four portions during 10 min. The reaction mixture was stirred over night at room temperature. Then most of the methanol was removed in vacuo and to the residue was added 100 ml of water. A yellow solid was filtered, washed with cold water and dried. The crude product was crystallized with methanol.

The physical, analytical and spectral data of new *cis*-3-hydroxy-2-(1-phenyl-3-aryl-4-pyrazolyl) chromanone dimethylacetals (**2**) are given below.

***cis*-3-Hydroxy-2-(1, 3-diphenyl-4-pyrazolyl) chromanone dimethylacetal (2a).** Yield 71%; mp 154-156°C; IR (KBr): 3476 cm<sup>-1</sup> (-OH str.); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300MHz): δ 2.18 (d, 1H, OH, *J* = 5.1 Hz), 4.21 (d, 1H, C<sub>3</sub>-H, *J* = 5.1 Hz), 5.53 (bs, 1H, C<sub>2</sub>-H), 3.21 (s, 3H, OCH<sub>3</sub>), 3.47 (s, 3H, OCH<sub>3</sub>), 8.56 (s, 1H), 7.81 (m, 5H), 7.34-7.48 (m, 7H), 7.02-7.05 (m, 2H); Anal. Cald. for C<sub>26</sub>H<sub>24</sub>N<sub>2</sub>O<sub>4</sub>: C, 72.90, H, 5.61, N, 6.54. Found: C, 73.02, H, 5.78, N, 6.56; MS (m/z): 429 (M<sup>+</sup>+1, base peak), 428 (M<sup>+</sup>), 397, 262, 167, 166.

***cis*-3-Hydroxy-2-[(1-phenyl-3-(*p*-tolyl)-4-pyrazolyl)] chromanone dimethylacetal (2b).** Yield 74%; mp 178-180°C; IR (KBr): 3494 cm<sup>-1</sup> (-OH str.); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300MHz): δ 2.17 (d, 1H, OH, *J* = 5.1 Hz), 4.20 (d, 1H, C<sub>3</sub>-H, *J* = 5.1 Hz), 5.52 (bs, 1H, C<sub>2</sub>-H), 2.42 (s, 3H, CH<sub>3</sub>), 3.22 (s, 3H, OCH<sub>3</sub>), 3.47 (s, 3H, OCH<sub>3</sub>), 8.55 (s, 1H), 7.83 (d, 2H, *J* = 7.8 Hz), 7.74 (d, 2H, *J* = 7.8 Hz), 7.62 (dd, 1H), 7.44-7.50 (m, 2H), 7.28-7.33 (m, 4H), 7.01-7.04 (m, 2H); Anal. Cald. for C<sub>27</sub>H<sub>26</sub>N<sub>2</sub>O<sub>4</sub>: C, 73.30, H, 5.88, N, 6.33. Found: C, 73.18, H, 6.04, N, 6.51.

***cis*-3-Hydroxy-2-[(1-phenyl-3-(*p*-anisyl)-4-pyrazolyl)] chromanone dimethylacetal (2c).** Yield 75%; mp 172-173°C; IR (KBr): 3513 cm<sup>-1</sup> (-OH str.); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300MHz): δ 2.17 (d, 1H, OH, *J* = 5.1 Hz), 4.20 (d, 1H, C<sub>3</sub>-H, *J* = 5.1 Hz), 5.52 (bs, 1H, C<sub>2</sub>-H), 3.89 (s, 3H, OCH<sub>3</sub>), 3.22 (s, 3H, OCH<sub>3</sub>), 3.46 (s, 3H, OCH<sub>3</sub>), 8.54 (s, 1H), 7.90 (d, 2H, *J* = 7.8 Hz), 6.97 (d, 2H, *J* = 7.8 Hz), 7.64 (dd, 1H), 7.37-7.55 (m, 6H), 7.01-7.05 (m, 2H); Anal. Cald. for C<sub>27</sub>H<sub>26</sub>N<sub>2</sub>O<sub>5</sub>: C, 70.74, H, 5.67, N, 6.11. Found: C, 70.79, H, 5.79, N, 6.28; MS (m/z): 459 (M<sup>+</sup>+1, base peak), 458 (M<sup>+</sup>), 427, 292, 167, 166.

***cis*-3-Hydroxy-2-[(1-phenyl-3-(*p*-chlorophenyl)-4-pyrazolyl)] chromanone dimethyl acetal (2d).** Yield 69%; mp 167-168°C; IR (KBr): 3487 cm<sup>-1</sup> (-OH str.); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300MHz): δ 2.19 (d, 1H, OH, *J* = 5.1 Hz), 4.20 (d, 1H, C<sub>3</sub>-H, *J* = 5.1 Hz), 5.49 (bs, 1H, C<sub>2</sub>-H), 3.23 (s, 3H, OCH<sub>3</sub>), 3.48 (s, 3H, OCH<sub>3</sub>), 8.56 (s, 1H), 7.88 (d, 2H, *J* = 8.4 Hz), 7.68 (d, 2H, *J* = 8.4 Hz), 7.60 (dd, 1H), 7.41-7.49 (m, 2H), 7.31-7.35 (m, 4H), 7.03-7.08 (m, 2H); Anal. Cald. for C<sub>26</sub>H<sub>23</sub>N<sub>2</sub>O<sub>4</sub>Cl: C, 67.53, H, 4.98, N, 6.06. Found: C, 67.45, H, 5.11, N, 6.09.

***cis*-3-Hydroxy-2-[(1-phenyl-3-(*p*-fluorophenyl)-4-pyrazolyl)] chromanone dimethyl acetal (2e).** Yield 65%; mp 163-165°C; IR (KBr): 3493 cm<sup>-1</sup> (-OH str.); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300MHz): δ 2.18 (d, 1H, OH, *J* = 5.1 Hz), 4.19 (d, 1H, C<sub>3</sub>-H, *J* = 5.1 Hz), 5.46 (bs, 1H, C<sub>2</sub>-H), 3.22 (s, 3H, OCH<sub>3</sub>), 3.47 (s, 3H, OCH<sub>3</sub>), 8.55 (s, 1H), 7.83-7.95 (m, 4H), 7.45-7.66 (m, 5H), 7.13-7.16 (m, 2H), 7.01-7.04 (m, 2H); Anal. Cald. for C<sub>26</sub>H<sub>23</sub>N<sub>2</sub>O<sub>4</sub>F: C, 69.95, H, 5.16, N, 6.28. Found: C, 70.06, H, 5.18, N, 6.41.

***cis*-3-Hydroxy-2-[(1-phenyl-3-(*p*-nitrophenyl)-4-pyrazolyl)] chromanone dimethyl acetal (2f).** Yield 66%; mp 151-153°C; IR (KBr): 3466 cm<sup>-1</sup> (-OH str.); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300MHz): δ 2.21 (d, 1H, OH, *J* = 5.1 Hz), 4.22 (d, 1H, C<sub>3</sub>-H, *J* = 5.1 Hz), 5.49 (bs, 1H, C<sub>2</sub>-H), 3.24 (s, 3H, OCH<sub>3</sub>), 3.48 (s, 3H, OCH<sub>3</sub>), 8.59 (s, 1H), 8.33 (d, 2H, *J* = 9.0 Hz), 8.08 (d, 2H, *J* = 9.0 Hz), 7.79-7.83 (m, 2H), 7.33-7.48 (m, 5H), 7.01-7.07 (m, 2H); Anal. Cald. for C<sub>26</sub>H<sub>23</sub>N<sub>3</sub>O<sub>6</sub>: C, 65.96, H, 4.86, N, 8.88. Found: C, 66.04, H, 4.99, N, 9.01.

**General procedure for preparation of *trans*-3-hydroxy-2-(1-phenyl-3-aryl-4-pyrazolyl) chromanones (3)**

A solution (0.002 mol) of dimethylacetals (2) in 10 ml of acetone was treated with 2 ml of concentrated hydrochloric acid. The resulting solution was kept for 2 hours at room temperature. During this time a white crystalline product separated. The product was filtered, washed with cold acetone and dried. The crude product was crystallized with methanol.

The physical, analytical and spectral data of new 3-*trans*-hydroxy-2-(1-phenyl-3-aryl-4-pyrazolyl) chromanones (3) are given below:

***trans*-3-Hydroxy-2-(1, 3-diphenyl-4-pyrazolyl) chromanone (3a).** Yield 80%; mp 222-224°C; IR (KBr): 3375 cm<sup>-1</sup> (-OH str.), 1679 cm<sup>-1</sup> (C=O str.); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300MHz): δ 4.83 (d, 1H, C<sub>3</sub>-H, *J* = 12.6 Hz), 5.35 (d, 1H, C<sub>2</sub>-H, *J* = 12.6 Hz), 3.80 (bs, 1H, OH), 8.31 (s, 1H), 7.71-7.94 (m, 7H), 7.47-7.59 (m, 5H), 7.07-7.14 (m, 2H); Anal. Cald. for C<sub>24</sub>H<sub>18</sub>N<sub>2</sub>O<sub>3</sub>: C, 75.39, H, 4.71, N, 7.33. Found: C, 75.35, H, 4.85, N, 7.45; MS (m/z): 383 (M<sup>+</sup>+1, base peak), 382 (M<sup>+</sup>), 353, 262, 121, 120, 107.

***trans*-3-Hydroxy-2-[(1-phenyl-3-(*p*-tolyl)-4-pyrazolyl)] chromanone (3b).** Yield 82%; mp 239-241°C; IR (KBr): 3370 cm<sup>-1</sup> (-OH str.), 1684 cm<sup>-1</sup> (C=O str.); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300MHz): δ 2.39 (s, 3H, CH<sub>3</sub>), 4.82 (d, 1H, C<sub>3</sub>-H, *J* = 12.6 Hz), 5.33 (d, 1H, C<sub>2</sub>-H, *J* = 12.6 Hz), 3.79 (bs, 1H, OH), 8.31 (s, 1H), 7.96 (dd, 1H), 7.82 (d, 2H, *J* = 8.4 Hz), 7.71 (d, 2H, *J* = 8.4 Hz), 7.47-7.63 (m, 6H), 7.06-7.14 (m, 2H); Anal. Cald. for C<sub>25</sub>H<sub>20</sub>N<sub>2</sub>O<sub>3</sub>: C, 75.76, H, 5.05, N, 7.07. Found: C, 75.87, H, 5.16, N, 7.17.

***trans*-3-Hydroxy-2-[(1-phenyl-3-(*p*-anisyl)-4-pyrazolyl)] chromanone (3c).** Yield 78%; mp 235-236°C; IR (KBr): 3378 cm<sup>-1</sup> (-OH str.), 1679 cm<sup>-1</sup> (C=O str.); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300MHz): δ 3.84 (s, 3H, OCH<sub>3</sub>), 4.83 (d, 1H, C<sub>3</sub>-H, *J* = 12.6 Hz), 5.34 (d, 1H, C<sub>2</sub>-H, *J* = 12.6 Hz), 3.65 (bs, 1H, OH), 8.30 (s, 1H), 7.97 (dd, 1H), 7.78 (d, 2H, *J* = 8.4 Hz), 7.02 (d, 2H, *J* = 8.4 Hz), 7.48-7.62 (m, 6H), 7.07-7.17 (m, 2H); Anal. Cald. for C<sub>25</sub>H<sub>20</sub>N<sub>2</sub>O<sub>4</sub>: C, 72.81, H, 4.85, N, 6.80. Found: C, 72.71, H, 4.92, N, 6.91; MS (m/z): 413 (M<sup>+</sup>+1, base peak), 412 (M<sup>+</sup>), 383, 292, 121, 120, 107.

***trans*-3-Hydroxy-2-[(1-phenyl-3-(*p*-chlorophenyl)-4-pyrazolyl)] chromanone (3d).** Yield 72%; mp 230-231°C; IR (KBr): 3375 cm<sup>-1</sup> (-OH str.), 1680 cm<sup>-1</sup> (C=O str.); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300MHz): δ 4.84 (d, 1H, C<sub>3</sub>-H, *J* = 12.6 Hz), 5.37 (d, 1H, C<sub>2</sub>-H, *J* = 12.6 Hz), 3.75 (bs, 1H, OH), 8.13 (s, 1H), 7.98 (dd, 1H), 7.91 (d, 2H, *J* = 8.4 Hz), 7.71 (d, 2H, *J* = 8.4 Hz), 7.50-7.65 (m, 6H), 7.06-7.15 (m, 2H); Anal. Cald. for C<sub>24</sub>H<sub>17</sub>N<sub>2</sub>O<sub>3</sub>Cl: C, 69.23, H, 4.09, N, 6.73. Found: C, 69.34, H, 4.17, N, 6.89.

***trans*-3-Hydroxy-2-[(1-phenyl-3-(*p*-fluorophenyl)-4-pyrazolyl)] chromanone (3e).** Yield 74%; mp 225-226°C; IR (KBr): 3371 cm<sup>-1</sup> (-OH str.), 1682 cm<sup>-1</sup> (C=O str.); <sup>1</sup>H NMR (CDCl<sub>3</sub>,

300MHz):  $\delta$  4.83 (d, 1H, C<sub>3</sub>-H,  $J$  = 12.6 Hz), 5.35 (d, 1H, C<sub>2</sub>-H,  $J$  = 12.6 Hz), 3.72 (bs, 1H, OH), 8.15 (s, 1H), 7.51-7.91 (m, 9H), 7.06-7.22 (m, 4H); Anal. Cald. for C<sub>24</sub>H<sub>17</sub>N<sub>2</sub>O<sub>3</sub>F: C, 72.00, H, 4.25, N, 7.00. Found: C, 72.11, H, 4.18, N, 7.05; MS (m/z): 401 (M<sup>+</sup>+1, base peak), 400 (M<sup>+</sup>), 371, 280, 121, 120, 107.

**trans-3-Hydroxy-2-[(1-phenyl-3-(p-nitrophenyl)-4-pyrazolyl)] chromanone (3f).** Yield 70%; mp 237-238°C; IR (KBr): 3368 cm<sup>-1</sup> (-OH str.), 1685 cm<sup>-1</sup> (C=O str.); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300MHz):  $\delta$  4.83 (d, 1H, C<sub>3</sub>-H,  $J$  = 12.6 Hz), 5.35 (d, 1H, C<sub>2</sub>-H,  $J$  = 12.6 Hz), 3.62 (bs, 1H, OH), 8.31 (s, 1H), 7.96 (dd, 1H), 8.37 (d, 2H,  $J$  = 8.4 Hz), 8.13 (d, 2H,  $J$  = 8.4 Hz), 7.45-7.59 (m, 6H), 7.06-7.18 (m, 2H); Anal. Cald. for C<sub>24</sub>H<sub>17</sub>N<sub>3</sub>O<sub>5</sub>: C, 67.45, H, 3.98, N, 9.84. Found: C, 67.47, H, 4.02, N, 9.88.

**General procedure for preparation of cis-3-hydroxy-2-(1-phenyl-3-aryl-4-pyrazolyl) chromanones (4).** A mixture of dimethylacetals **2** (0.005 mol) and 40 ml of 50% acetic acid was stirred at 60-70 °C for 6 hours. The resulting solution after cooling was mixed with 40 ml of water and the solid was filtered, washed with cold water and dried. The crude product was crystallized with methanol.

**cis-3-Hydroxy-2-(1, 3-diphenyl-4-pyrazolyl) chromanone (4a).** Yield 51%; mp 198-199°C; IR (KBr): 3338 cm<sup>-1</sup> (-OH str.), 1689 cm<sup>-1</sup> (C=O str.); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300MHz):  $\delta$  8.34 (s, 1H), 3.55 (bs, 1H, OH), 4.68 (d, 1H, C<sub>3</sub>-H,  $J$  = 5.1 Hz), 5.56 (d, 1H, C<sub>2</sub>-H,  $J$  = 5.1 Hz), 7.56-7.91 (m, 12H), 7.12-7.17 (m, 2H); Anal. Cald. for C<sub>24</sub>H<sub>18</sub>N<sub>2</sub>O<sub>3</sub>: C, 75.39, H, 4.71, N, 7.33. Found: C, 75.41, H, 4.79, N, 7.39; MS (m/z): 383 (M<sup>+</sup>+1, base peak), 382 (M<sup>+</sup>), 353, 262, 121, 120, 107.

**cis-3-Hydroxy-2-[(1-phenyl-3-(p-tolyl)-4-pyrazolyl)] chromanone (4b).** Yield 55%; mp 204-206°C; IR (KBr): 3392 cm<sup>-1</sup> (-OH str.), 1687 cm<sup>-1</sup> (C=O str.); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300MHz):  $\delta$  2.35 (s, 3H, CH<sub>3</sub>), 8.35 (s, 1H), 3.47 (bs, 1H, OH), 4.66 (d, 1H, C<sub>3</sub>-H,  $J$  = 5.1 Hz), 5.57 (d, 1H, C<sub>2</sub>-H,  $J$  = 5.1 Hz), 7.86 (d, 2H,  $J$  = 8.4 Hz), 7.71 (d, 2H,  $J$  = 8.4 Hz), 7.41-7.67 (m, 7H), 7.10-7.18 (m, 2H); Anal. Cald. for C<sub>25</sub>H<sub>20</sub>N<sub>2</sub>O<sub>3</sub>: C, 75.76, H, 5.05, N, 7.07. Found: C, 75.92, H, 5.07, N, 7.09.

## Acknowledgements

We are thankful to CSIR, New Delhi for the award of Senior Research Fellowship to Rajesh Kumar and DRDO, New Delhi (Grant No ERIP/ER/0303447/M/01) to carry out this work. Thanks are also due to RSIC, CDRI, Lucknow, India, for providing mass and elemental analysis.

## References and Notes

- (a) Varvoglis, A. *The Organic Chemistry of Polycordinated Iodine*; VCH: New York, 1992.  
(b) Varvoglis, A. *Hypervalent Iodine in Organic Synthesis*; Academic: London, 1997. (c) Zhdankin, V. V.; Stang, P. J. *Chem. Rev.* **2002**, *102*, 2523.

2. (a) Varoglis, A. *Chem. Soc. Rev.* **1981**, *10*, 377. (b) Moriarty, R. M.; Prakash, O. *Acc. Chem. Res.* **1986**, *19*, 244. (c) Ramsden, C. A. *Chem. Soc. Rev.* **1994**, *23*, 111.
3. (a) Moreno, I.; Tellitu, I.; Etayo, J.; SanMartin, R.; Domenguez, E. *Tetrahedron* **2001**, *57*, 5403. (b) Kita, Y.; Takada, T. *J. Org. Chem.* **1995**, *60*, 6499.
4. (a) Moriarty, R. M.; Vaid, R. K.; Koser, G. F. *Synlett* **1990**, 365. (b) Koser, G. F. *Aldrichim. Acta* **2001**, *34*, 89.
5. (a) Wirth, T. *Angew. Chem. Int. Ed.* **2001**, *40*, 2812. (b) Kuhakarn, C.; Kittigowittana, K.; Pohmakotr, M.; Reutrakul, V. *Arkivoc* **2005(i)**, 143. (c) De Munari, S.; Frigerio, M.; Santagostino, M. *J. Org. Chem.* **1996**, *61*, 9272. (d) Nicolaou, K. C.; Mathison, C. J. N.; MontaGnon, T. *J. Am. Chem. Soc.* **2004**, *126*, 5192. (e) Zhdankin, V. V.; Koposov, A. Y.; Netzel, B. C.; Yashin, N. V.; Rempel, B. P.; Freguson, M. J.; Tykwinski, R. R. *Angew. Chem. Int. Ed.* **2003**, *42*, 2194.
6. (a) Zhdankin, V. V.; Stang, P. J. *Chem. Rev.* **2002**, *102*, 2523. (b) Speicher, A.; Bomm, V.; Eicher, T. *J. Prakt. Chem.* **1996**, *338*, 588. (c) Chaudhari, S. S. *Synlett* **2000**, 278; (d) Kumamoto, H.; Ogamino, J.; Tanaka, H.; Suzuki, H.; Haraguchi, K.; Miyasaka, T.; Yokomatsu, T.; Shibuya, S. *Tetrahedron* **2001**, *57*, 3331. (e) Promarak, V.; Burn, P. L. *J. Chem. Soc. Perkin Trans I* **2001**, 14.
7. Prakash, O. *Aldrichim. Acta* **1995**, *28*, 63.
8. Prakash, O.; Pahuja, S.; Moriarty, R. M. *Synth. Commun.* **1990**, *20*, 1417.
9. Moriarty, R. M.; Prakash, O.; Mussallam, H. A.; Mahesh, V. K. *Heterocycles*, **1986**, *24*, 1641.
10. Prakash, O.; Pahuja, S.; Goyal, S.; Sawhney, S. N.; Moriarty, R. M. *Synlett* **1990**, 337.
11. Prakash, O.; Pahuja, S.; Tanwar, M. P. *Indian J. Chem.* **1994**, *34B*, 272.
12. Prakash, O.; Pundeer, R.; Kaur, H. *Synthesis* **2003**, *18*, 2768.
13. Prakash, O.; Kaur, H.; Sharma, V.; Bhardwaj, V.; Pundeer, R. *Tetrahedron Lett.* **2004**, *45*, 9065.
14. Prakash, O.; Tanwar, M. P. *Bull. Chem. Soc. Jpn.* **1995**, *68*, 1168.
15. Moriarty, R. M.; Prakash, O. *J. Org. Chem.* **1985**, *50*, 151
16. Prakash, O.; Pahuja, S.; Goyal, S.; Sawhney, S. N. *Indian J. Chem.* **1991**, *30B*, 1023.
17. Prakash, O.; Mendiratta, S. *Synth. Commun.* **1992**, *22*, 327.
18. (a) Jung, J. C.; Walkins, E. B.; Avery, M. A. *Tetrahedron* **2002**, *58*, 3039. (b) Kane, J. L.; Hirth, B. H.; Laing, O.; Gourlie, B. B.; Nahill, S.; Barsomiam, G. *Bioorg. Med. Chem. Lett.* **2003**, *13*, 4463. (c) Rathelot, P.; Azas, N.; El-Kashef, H.; Delmas, F.; Di Giorgio, C.; Timon-David, P.; Maldonado, J.; Vanelle, P. *Eur. J. Med. Chem.* **2002**, *37*, 671.
19. (a) Ferry, D. R.; Smith, A.; Malkhandi, J.; Fyfe, D. W.; deTakats, P. G.; Anderson, D.; Baker, J.; Kerr, D. J. *Clin. Cancer Res.* **1996**, *2*, 659. (b) Chiang, L. C.; Chiang, W.; Liu, M. C.; Lin, C. C. *J. Antimicrobial Chem.* **2003**, *52*, 194. (c) Kumazawa, S.; Hamasaka, T.; Nakayama, T. *Food Chem.* **2004**, *84*, 329.
20. Buset, H.; Scheline, R. R. *Biomed. Mass Spect.* **1979**, *6*, 213.
21. Blatt, A. H. *Org. Synth. Coll. Vol. II* **1959**, 304.