ISSN: 2249 –4820

Chemistry & Biology Interface, 2013, 3, 5, 314-333

Chemistry & Biology Interface

An official Journal of ISCB, Journal homepage; www.cbijournal.com

Research Paper

Convenient Synthesis of Some New Diversely Functionalized Coumarinyl Chalcone Derivatives under Microwaves

[a]Vishwa Dhinoja, [a]Vicky Jain, [a]Shailesh Thakrar, [a]Jayant Rathod, [a]Mitesh Chhatrola, [b]Disha Tilala, [c]Denish Karia and [a]Anamik Shah*

[a]*Department of Chemistry, Saurashtra University, Rajkot-360005, Gujarat (India).*
[b]*Department of Chemistry, St. Xavier's College, Ahmedabad-380009 (Gujarat) India.*
[c]*Patel J. D. K. Dovelwala Science College, Borsad-388540 (Anand), Gujarat (India).*
Received 24 August 2013; Accepted 4October 2013

Keywords: Chalcones, 3-Acetyl 4-hydroxy coumarin, Pyrazole aldehyde, Chloroform, Piperidine.

Abstract: Herein we have reported some new coumarinyl chalcones synthesized by conventional method as well as microwave assisted method of synthesis. The reaction of 3-acetyl 4-hydroxy coumarin **2a-c** with substituted 1, 3-diphenyl-1H-pyrazole-4-carbaldehyde **6a-h** in the presence of piperidine as catalyst and chloroform as a solvent, yielded a series of chalcones **7a-x**. The structures of all synthesized compounds are well characterized by Mass, FT-IR, ^{1}H NMR, ^{13}C NMR and elemental analysis. After obtaining experimental data regarding the yield and the time taken for the synthesis by both the methods, convenient and microwave assisted method, it was proved that the microwave assisted method is convenient for synthesis of this type of coumarinyl chalcones **7a-x**.

Introduction

Coumarin derivatives constitute an important class of compounds with a wide range of biological activities [1-3]. They have been reported to exhibit antitumor [4], antioxidant [5] and anti-inflammatory [6] properties. Recently, Musa et al. have reviewed the application of potential coumarin derivatives in the pharmacotherapy of breast cancer [7]. Also, Lee et al. have reported the isolation of a coumarin containing compound, showing significant inhibition activity against two ER + human breast cancer cell lines [8-9]. Some phenyl coumarins have been proposed as suppressors of LTR-dependent transcription [10]. A natural dipyranocoumarin, (+)-Calanolide A, is also under anti-AIDS clinical trials [11]. Using 4-hydroxycoumarins, reported for antibacterial and antioxidant activities [12-13], we have synthesized 3-Acetyl, 4-hydroxy coumarins by a reported method [14]. On the other hand, pyrazole skeleton, found in many biologically active compounds, represents an interesting template for combinatorial [15] as well as medicinal chemistry [16]. Pyrazole derivatives have been found to

Corresponding Author* E-mail:anamik_shah@hotmail.com

ISSN: 2249 –4820
Chemistry & Biology Interface, 2013, 3, 5, 314-333

have antimalarial [17], antitubercular [18], anticonvulsant [19], anti-inflammatory [20], antimicrobial [21] and antihyperglycemic activity [22]. Certain alkyl pyrazoles show significant bacteriostatic, bactericidal and fungicidal, analgesic and antipyretic activities. A hybridization of two biologically active pharmacophoric groups is a promising area for new drug discovery. The hybrid may have significant biological profile compared to the corresponding active pharmacophore. Chalcones are an important class of compounds widely distributed in nature and considered as the precursors for flavonoid synthesis in plants [23]. In recent years, chemistry of chalcones has fascinated interest as these compounds have been found to exhibit several biological activities, such as cytotoxic [24], antimalarial [25], antileishmanial [26], anti-inflammatory [27], anti-HIV [28], and as tyrosinekinase inhibitors [29]. The most important naturally occurring chalcone, licochalcone-A, was identified as a potential drug candidate against leishmania, trypanosoma and plasmodium parasites [30].

Chemistry

The synthesis of coumarinyl chalcone (**7a-x**) is depicted in Scheme 3. The key intermediate 3-acetyl,4-hydroxy-coumarins (**2a-c**) was prepared using 4-hydroxycoumarins (**1a-c**) by reported method [11] (Scheme 1). 4-Formyl pyrazoles (**6a-h**) were prepared by Vilsmeier-Haack reaction of hydrazones (**5a-h**) (Scheme 2). The final product (**7a-x**) was prepared by reacting **2a-c** with **6a-h** using conventional method as well as microwave as shown in Scheme 3.

Materials and Methods

Melting points of all the synthesized compounds have been recorded by open capillary method. Formation of the compounds was routinely checked by TLC on silica gel-G plates of 0.5 mm thickness and spots were located by iodine and UV. All the reactions were carried out in **Q-pro-M** microwave synthesizer. The IR spectra were recorded on a **Shimadzu FT-IR-8400** instrument using KBr pellet method. Mass spectra were recorded on Shimadzu **GC-MS-QP-2010** model using Direct Injection Probe technique. ^{1}H NMR was determined in $CDCl_3$/DMSO solution on a **Bruker Ac 400 MHz** spectrometer. Elemental analysis of the all the synthesized compounds were carried out on Elemental **Vario EL III Carlo Erba 1108** model and the results are in agreements with the structures assigned.

Experimental Procedure

General method for preparation of 3-acetyl 4-hydroxy coumarin (2a-c):

To a solution of 4-hydroxy coumarin **1a-c** (0.017 mol) in glacial acetic acid (15 ml), phosphorous oxychloride (14ml) was added drop wise at 0^{o}C, the resulting mixture was refluxed for 2-3 hrs and then poured on crushed ice with stirring. The solid separated out was filtered, washed with water and crystallized from methanol to give 3-acetyl 4-hydroxy coumarin **2a-c** as solid. Yield - 65%, M.P. - 117^{o}C.

General method for preparation of acetophenone phenyl hydrazones (5a-h):

Phenyl hydrazine (0.1 mol) was added to a solution of substituted acetophenone **4a-h** (0.1 mol) in ethanol (50 ml) along with 3-4 drops of glacial acetic acid. The resulting mixture was stirred for 1 hour at room temperature. The progress and the completion of reaction were monitored by thin layer chromatography using ethyl acetate: hexane (6:4) as a mobile phase.

ISSN: 2249 –4820

Chemistry & Biology Interface, 2013, 3, 5, 314-333

After the completion of reaction, the reaction mixture was kept at room temperature for 1 hours and the crystalline product was collected by filtration. The product was washed with ethanol and dried to give substituted acetophenone phenyl hydrazone in good yield which was pure enough to use for the next step.

General method for preparation of pyrazole aldehydes (6a-h):

Phosphorous oxychloride (0.032 mol) was added drop wise to a 25 ml flat bottom flask containing Dimethylformamide (0.32 mol) under stirring at 0-5^{o}C. After the completion of addition, the mixture was stirred at this temperature for 10-15 min. Freshly prepared acetophenone phenyl hydrazone **5a-h** (0.03 mol) was added to above mixture and the content was heated on water bath for 5-6 hours. The progress and the completion of reaction were monitored by thin layer chromatography using toluene: ethyl acetate (6: 4) as a mobile phase. After the reaction to be completed, the reaction mixture was cooled to room temperature and the content of the flask was poured on crushed ice to isolate the product. The separated product was filtered off and washed with cold water to remove acidity. It was dried at 65^{o}C and recrystallized by methanol to give crystalline pyrazole aldehydes **6a-h** in good yield.

General method for preparation of 3-acetyl, 4-hydroxy coumarinyl chlcones (7a-x):

A mixture of 3-acetyl 4-hydroxy coumarin 2**a-c** (0.01 mol) and) substituted pyrazole aldehyde **6a-h** (0.01 mol) were dissolved in 30 ml of solvent (**Table 1**). The catalytic amount of piperidine (0.02 ml) was added and the reaction mixture was subjected to microwave for a specific time at lower power (350W). The progress of the reaction was monitored by TLC examination at an interval of every minute ethyl acetate: hexane (2:3). The reaction was carried out using different solvents. The time (in minutes) taken for the completion of reaction and the % yield were observed (**Table 1**). Since the use of chloroform as solvent resulted in highest yield within shortest time, the same reaction was performed using chloroform as solvent and piperidine as catalyst at reflux temperature in a round bottom flask to carry out the reaction in conventional way. On the completion of reaction, the excess of chloroform was distilled out and the resulting mass was cooled and titurated with methanol. The solid separated was filtered and washed with methanol, dried and then yield obtained in a particular time (in hours) were calculated. The comparative data obtained regarding the yields by microwave assisted as well as conventional method of synthesis for the compounds **7a-x** are shown in **Table 3**.

Result and Discussion

Initially, we have synthesized **7a, 7i** and **7q** in different solvents under microwave irradiation. We observed the higher yield within lesser time in the reaction which was carried out using Chloroform as solvent, instead of Dichloromethane, DMF and Ethyl Acetate. The time taken for the completion of reaction was 3-9 minutes and the % yields observed was 70-85% under microwave assisted method. Then we carried out the same synthesis using chloroform as solvent and piperidine as catalyst under conventional method. The time taken for synthesis of all **7a-x** compounds by conventional method was about 4-5 hours and the % yield observed was about 48-68%. All the synthesized compounds were characterized by TLC, Melting point, elemental analysis, IR ^{1}H NMR and ^{13}C

ISSN: 2249 –4820

Chemistry & Biology Interface, 2013, 3, 5, 314-333

NMR. Elemental analysis indicated the % of the elements very close to the theoretical values.

Conclusion

In conclusion, the microwave assisted procedure is expedient for the synthesis of highly functionalized chalcone derivatives. The merits of procedure are efficient methodology, excellent yields, cleaner reaction profiles, simple work-up and enormous number of functional group compatibility.

Spectral Data

(E)-3-(3-(1,3-diphenyl-1H-pyrazol-4-yl)acryloyl)-4-hydroxy-6-methyl-2H-chromen-2-one (7a)

M.P. 218-220°C. IR (KBr) cm^{-1}: 3606, 3540, 3026, 2980, 2891, 1717, 1479, 1443, 1371, 1282, 1215, 998, 962, 704, ^{1}H NMR 400 MHz: (DMSO-d_6, δ ppm): 2.32-2.34 (s, 3H), 7.03-7.05 (d, 2H), 7.30-7.32 (d, 1H), 7.44-7.46 (dd, 3H), 7.50-7.52 (dd, 2H), 7.58-7.60 (m, 3H), 7.79-7.81 (d, 2H), 7.89-7.91 (dd, 1H), 8.24-8.26 (s, 1H), ^{13}C NMR 400 MHz: (DMSO-d_6, δ ppm): 21.7, 98.9, 113.0, 117.3, 119.9, 126.2, 127.0, 127.5, 128.7, 129.2, 129.3, 131.4, 132.0, 133.0, 135.1, 139.7, 149.5, 152.6, 159.4, 179.9, 183.7. Mass: [m/e (%)], M. Wt.: 448.14 anal. Calcd. for $C_{28}H_{20}N_2O_4$: C, 74.99; H, 4.50; N, 6.25; O, 14.27 Found: C, 74.49; H, 4.45; N, 6.20; O, 14.20%.

(E)-4-hydroxy-6-methyl-3-(3-(1-phenyl-3-(p-tolyl)-1H-pyrazol-4-yl)acryloyl)-2H-chromen-2-one (7b)

M.P. 172-174°C. IR (KBr) cm^{-1}:3616, 3560, 3036, 2981, 2892, 1707, 1489, 1453, 1372, 1281, 1246, 1205, 997, 961, 735. ^{1}H NMR 400 MHz: (DMSO-d_6, δ ppm): 2.35-2.36 (s, 6H), 7.05-7.09 (d, 2H), 7.28-7.30 (m, 3H), 7.44-7.46 (dd, 2H), 7.50-7.52 (dd, 3H), 7.58-7.60 (dd, 2H), 7.67-7.69 (dd, 2H), 8.40-8.42 (s, 1H), ^{13}C NMR 400 MHz: (DMSO-d_6, δ ppm): 21.3, 21.7, 98.9, 113.0, 116.9, 117.3, 119.9, 126.2, 127.0, 128.4, 129.3, 130.0, 131.4, 131.7, 132.0, 135.1, 139.7, 149.5, 150.4, 152.6, 159.4, 179.9, 183.3. Mass: [m/e (%)], M. Wt.: 462.16 anal. Calcd. for $C_{29}H_{22}N_2O_4$: C, 75.31; H, 4.79; N, 6.06; O, 13.84 Found: C, 75.26; H, 4.65; N, 5.90; O, 13.64 %.

(E)-4-hydroxy-6-methyl-3-(3-(3-(4-nitrophenyl)-1-phenyl-1H-pyrazol-4-yl)acryloyl)-2H-chromen-2-one (7c)

M.P. 230-232°C. IR (KBr) cm^{-1}: 3618, 3514, 3120, 3041, 2947, 2835, 1730, 1622, 1512, 1408, 1344, 1286, 1219, 1099, 856, 754, ^{1}H NMR 400 MHz: (DMSO-d_6, δ ppm): 2.37-2.39 (s, 3H), 7.14-7.16 (d, 1H), 7.37-7.39 (t, 1H),7.49-7.51 (m, 3H), 7.77-7.79 (d, 2H), 7.80-7.82 (d, 1H), 7.83-7.85 (d, 2H), 8.03-8.05 (d, 1H), 8.26-8.28 (d, 1H), 8.30-8.32 (d, 2H), 8.49-8.51 (s, 1H), ^{13}C NMR 400 MHz: (DMSO-d_6, δ ppm): 21.7, 98.9, 116.9, 117.3, 119.9, 124.4, 126.2, 129.3, 132.0, 135.4, 139.7, 147.9, 149.5, 150.4, 152.6, 159.4, 179.9, 183.7. Mass: [m/e (%)], M. Wt.: 493.13 anal. Calcd. for $C_{28}H_{19}N_3O_6$: C, 68.15; H, 3.88; N, 8.52; O, 19.45 Found: C, 68.10; H, 5.78; N, 8.45; O, 19.40%.

(E)-3-(3-(3-(4-chlorophenyl)-1-phenyl-1H-pyrazol-4-yl)acryloyl)-4-hydroxy-6-methyl-2H-chromen-2-one (7d)

M.P. 212-214°C. IR (KBr) cm^{-1}: 3610, 3523, 3022, 2985, 2896, 1722, 1483, 1455, 1378, 1285, 1242, 1208, 988, 963, 738. ^{1}H NMR 400 MHz: (DMSO-d_6, δ ppm): 2.32-2.34 (s, 3H), 7.03-7.05 (d, 2H), 7.31-7.33 (m, 3H), 7.48-7.50 (dd, 2H), 7.52-7.54 (dd, 3H), 7.59-7.61 (dd, 2H), 7.67-7.69 (dd, 2H), 8.45-8.47 (s, 1H), ^{13}C NMR 400 MHz: (DMSO-d_6, δ ppm): 21.7, 98.9, 113.0, 116.9, 117.3, 119.9, 126.2, 127.0, 128.9, 129.3, 130.3, 131.4, 132.0, 134.3, 135.1, 139.7, 149.5, 152.6, 159.4, 179.9, 183.7. Mass: [m/e (%)], M. Wt.: 482.10 anal. Calcd. for

ISSN: 2249 –4820
Chemistry & Biology Interface, 2013, 3, 5, 314-333

$C_{28}H_{19}ClN_2O_4$: C, 69.64; H, 3.97; Cl, 7.34; N, 5.80; O, 13.25 Found: C, 69.62; H, 3.92; Cl, 7.30; N, 5.75; O, 13.16 %.

(E)-4-hydroxy-3-(3-(3-(3-hydroxyphenyl)-1-phenyl-1H-pyrazol-4-yl)acryloyl)-6-methyl-2H-chromen-2-one (7e)

M.P. 232-234°C. IR (KBr) cm^{-1}: 3620, 3549, 3086, 3036, 2914, 2872, 1710, 1620, 1558, 1444, 1400, 1292, 1174, 1093, 981, 823, 758, ^1H NMR 400 MHz: (DMSO-d_6, δ ppm): 2.37-2.39 (s, 3H), 7.00-7.10 (m, 2H), 7.32-7.34 (t, 1H), 7.44-7.47 (m, 4H), 7.58-7.60 (dd, 2H), 7.74-7.76 (d, 2H), 7.89-7.91 (d, 1H), 8.01-8.04 (dd, 1H), 8.20-8.24 (dd, 1H), 8.45-8.47 (s, 1H), ^{13}C NMR 400 MHz: (DMSO-d_6, δ ppm): 98.9, 113.0, 114.8, 115.9, 116.4, 119.9, 120.1, 123.3, 125.4, 126.2, 128.3, 129.3, 130.3, 130.6, 131.4, 134.4, 139.7, 150.4, 152.5, 157.5, 159.4, 179.9, 183.7. Mass: [m/e (%)], M. Wt.: 464.14 anal. Calcd. for $C_{28}H_{20}N_2O_5$: C, 72.41; H, 4.34; N, 6.03; O, 17.22 Found: C, 72.38; H, 4.24; N, 5.95 O, 17.12%.

(E)-4-hydroxy-6-methyl-3-(3-(3-(3-nitrophenyl)-1-phenyl-1H-pyrazol-4-yl)acryloyl)-2H-chromen-2-one (7f)

M.P. 228-230°C. IR (KBr) cm^{-1}: 3614, 3514, 3136, 3043, 2872, 2831, 1699, 1616, 1531, 1450, 1354, 1294, 1178, 1103, 815, 750 ^1H NMR 400 MHz: (DMSO-d6, δ ppm): 2.33-2.35 (s, 3H), 7.14-7.16 (d, 1H), 7.29-7.31 (dd, 1H),7.45-7.47 (dd, 2H), 7.55-7.57 (d, 1H), 7.57-7.59 (dd, 2H), 7.61-7.63 (dd, 2H), 7.76-7.78 (dd, 1H), 8.12-8.14 (d, 2H), 8.30-8.32 (d, 2H), 8.60-8.62 (s, 1H), ^{13}C NMR 400 MHz: (DMSO-d_6, δ ppm): 21.7, 98.9, 113.0, 116.9, 117.3, 119.9, 122.7, 123.9, 126.2, 127.0, 130.3, 131.4, 133.9, 135.1, 139.7, 148.4, 149.5, 152.6, 159.4, 179.9, 183.7. Mass: [m/e (%)], M. Wt.: 493.13 anal. Calcd. for $C_{28}H_{19}N_3O_6$: C, 68.15; H, 3.88; N, 8.52; O, 19.45 Found: C, 68.20; H, 3.83; N, 8.50; O, 19.39%.

(E)-4-hydroxy-3-(3-(3-(2-hydroxyphenyl)-1-phenyl-1H-pyrazol-4-yl)acryloyl)-6-methyl-2H-chromen-2-one (7g)

M.P. 224-226°C. IR (KBr) cm^{-1}: 3614, 3549, 3120, 3059, 2981, 2872, 1722, 1604, 1400, 1296, 1236, 1217, 1020, 856, 746, 678, ^1H NMR 400 MHz: (DMSO-d_6, δ ppm): 2.35-2.37 (s, 3H), 7.13-7.15 (s, 1H), 7.16-7.19 (t, 1H), 7.21-7.26 (m, 3H), 7.45-7.47 (d, 2H), 7.56-7.58 (m, 4H), 7.77-7.79 (d, 2H), 7.95-7.99 (d, 1H), 8.06-8.08 (d, 1H), 8.42-8.44 (s, 1H), ^{13}C NMR 400 MHz: (DMSO-d_6, δ ppm): 21.7, 98.9, 116.9, 117.3, 119.9, 120.5, 121.8, 126.2, 127.0, 130.3, 131.4, 132.0, 135.1, 139.7, 149.5, 150.4, 155.2, 159.4, 179.9, 183.7. Mass: [m/e (%)], M. Wt.: 464.14 anal. Calcd. for $C_{28}H_{20}N_2O_5$: C, 72.41; H, 4.34; N, 6.03; O, 17.22; Found: C, 72.29; H, 4.30; N, 5.95; O, 17.16%.

(E)-4-hydroxy-3-(3-(3-(2-methoxyphenyl)-1-phenyl-1H-pyrazol-4-yl)acryloyl)-6-methyl-2H-chromen-2-one (7h)

M.P. 220-222°C. IR (KBr) cm^{-1}: 3591, 3126, 3057, 2993, 2951, 2833, 1727, 1497, 1432, 1332, 1227, 1220, 1180, 1003, 908, 748, 684, ^1H NMR 400 MHz: (DMSO-d_6, δ ppm): 2.34-2.36 (s, 3H), 3.78-3.80 (s, 3H), 7.02-7.05 (dd, 2H), 7.11-7.13 (d, 1H), 7.29-7.31 (t, 1H), 7.44-7.50 (m, 5H), 7.58-7.61 (t, 3H), 7.89-7.91 (dd, 1H), 8.06-8.08 (dd, 1H), 8.43-8.45 (s, 1H), ^{13}C NMR 400 MHz: (DMSO-d_6, δ ppm): 21.7, 56.1, 98.9, 111.1, 113.0, 116.9, 117.3, 118.9, 119.9, 121.5, 126.2, 127.0, 129.3, 129.9, 130.3, 131.1, 131.4, 132.0 135.1, 149.5, 157.3, 179.9, 183.7. Mass: [m/e (%)], M. Wt.: 478.15 anal. Calcd. for $C_{29}H_{22}N_2O_5$: C, 72.79; H, 4.63; N, 5.85; O, 16.72 Found: C, 72.74; H, 4.28; N, 5.80; O, 16.70%.

(E)-3-(3-(1,3-diphenyl-1H-pyrazol-4-yl)acryloyl)-4-hydroxy-7-methyl-2H-chromen-2-one (7i)

M.P. 242-244°C. IR (KBr) cm^{-1}: 3606, 3540, 3026, 2980, 2891, 1717, 1479, 1443, 1371,

ISSN: 2249 –4820
Chemistry & Biology Interface, 2013, 3, 5, 314-333

1282, 1236, 1215, 998, 962, 704, ^{1}H NMR 400 MHz: (DMSO-d_6, δ ppm): 2.35-2.37 (s, 3H), 7.03-7.05 (d, 2H), 7.30-7.32 (d, 1H), 7.44-7.46 (dd, 3H), 7.50-7.52 (dd, 2H), 7.58-7.60 (m, 3H), 7.79-7.81 (d, 2H), 7.89-7.91 (dd, 1H), 8.24-8.26 (s, 1H), ^{13}C NMR 400 MHz: (DMSO-d_6, δ ppm): 21.3, 98.9, 111.8, 113.0, 117.4, 119.9, 125.7, 126.7, 127.5, 128.7, 129.2, 129.3, 130.3, 131.4, 133.0, 139.7, 143.1, 150.0, 150.4, 152.6, 159.4, 179.9, 183.7. Mass: [m/e (%)], M. Wt.: 448.47 anal. Calcd. for $C_{28}H_{20}N_2O_4$: C, 74.99; H, 4.50; N, 6.25; O, 14.27 Found: C, 74.97; H, 4.47; N, 6.20; O, 14.21 %.

(E)-4-hydroxy-7-methyl-3-(3-(1-phenyl-3-(p-tolyl)-1H-pyrazol-4-yl)acryloyl)-2H-chromen-2-one (7j)

M.P. 248-250°C. IR (KBr) cm^{-1}: 3616, 3560, 3036, 2981, 2892, 1707, 1489, 1453, 1372, 1281, 1246, 1205, 997, 961, 735, ^{1}H NMR 400 MHz: (DMSO-d_6, δ ppm): 2.35-2.36 (s, 6H), 7.05-7.09 (d, 2H), 7.28-7.30 (m, 3H), 7.44-7.46 (dd, 2H), 7.50-7.52 (dd, 3H), 7.58-7.60 (dd, 2H), 7.67-7.69 (dd, 2H), 8.40-8.42 (s, 1H), ^{13}C NMR 400 MHz: (DMSO-d_6, δ ppm): 21.3, 98.9, 111.8, 113.0, 117.4, 119.9, 125.7, 126.2, 126.7, 128.4, 129.3, 129.5, 130.0, 131.4, 131.7, 139.7, 143.1, 150.4, 152.6, 159.4, 179.9, 183.7. Mass: [m/e (%)], M. Wt.: 462.50 anal. Calcd. for $C_{29}H_{22}N_2O_4$: C, 75.31; H, 4.79; N, 6.06; O, 13.84 Found: C, 75.30; H, 4.77; N, 6.05; O, 13.80 %.

(E)-4-hydroxy-7-methyl-3-(3-(3-(4-nitrophenyl)-1-phenyl-1H-pyrazol-4-yl)acryloyl)-2H-chromen-2-one (7k)

M.P. 234-236°C. IR (KBr) cm^{-1}: 3585, 3525, 3591, 3091, 3039, 2949, 2877, 1708, 1614, 1518, 1446, 1344, 1288, 1230, 1143, 1030, 862, 744, 701, ^{1}H NMR 400 MHz: (DMSO-d_6, δ ppm): 2.34-2.36 (s, 3H), 7.15-7.17 (d, 1H), 7.34-7.36 (t, 1H), 7.49-7.51 (m, 3H), 7.75-7.77 (d, 2H), 7.82-7.84 (d, 1H), 7.85-7.87 (d, 2H), 8.05-8.07 (d, 1H), 8.24-8.26 (d, 1H), 8.30-8.32 (d, 2H), 8.45-8.47 (s, 1H), ^{13}C NMR 400 MHz: (DMSO-d_6, δ ppm): 21.3, 98.9, 111.8, 113.0, 117.4, 119.9, 124.4, 125.7, 126.2, 126.7, 129.3, 130.3, 131.4, 139.1, 139.7, 143.1, 147.9, 150.0, 150.4, 152.6, 159.5 179.9, 183.7. Mass: [m/e (%)], M. Wt.: 493.47 anal. Calcd. for $C_{28}H_{19}N_3O_6$: C, 68.15; H, 3.88; N, 8.52; O, 19.45 Found: C, 68.13; H, 3.85; N, 8.50; O, 19.43 %.

(E)-3-(3-(3-(4-chlorophenyl)-1-phenyl-1H-pyrazol-4-yl)acryloyl)-4-hydroxy-7-methyl-2H-chromen-2-one (7l)

M.P. 232-234°C. IR (KBr) cm^{-1}: 3616, 3560, 3036, 2981, 2892, 1707, 1489, 1372, 1281, 1246, 1205, 997, 961, 735, ^{1}H NMR 400 MHz: (DMSO-d_6, δ ppm): 2.32-2.34 (s, 3H), 7.03-7.05 (d, 2H), 7.31-7.33 (m, 3H), 7.48-7.50 (dd, 2H), 7.52-7.54 (dd, 3H), 7.59-7.61 (dd, 2H), 7.67-7.69 (dd, 2H), 8.45-8.47 (s, 1H), ^{13}C NMR 400 MHz: (DMSO-d_6, δ ppm): 21.3, 98.9, 111.8, 113.0, 117.4, 119.9, 125.7, 126.2, 126.7, 128.9, 129.3, 130.3, 131.1, 131.4, 134.3, 139.7, 143.1, 150.0, 152.6, 159.4, 179.9, 183.7. Mass: [m/e (%)], M. Wt.: 482.91 anal. Calcd. for $C_{28}H_{19}ClN_2O_4$: C, 69.64; H, 3.97; Cl, 7.34; N, 5.80; O, 13.25 Found: C, 69.63; H, 3.94; Cl, 7.33; N, 5.80; O, 13.22 %.

(E)-4-hydroxy-3-(3-(3-(3-hydroxyphenyl)-1-phenyl-1H-pyrazol-4-yl)acryloyl)-7-methyl-2H-chromen-2-one (7m)

M.P. 244-246°C. IR (KBr) cm^{-1}: 3630, 3556, 3093, 3064, 2982, 2887, 1705, 1614, 1516, 1427, 1292, 1232, 1095, 1024, 987, 958, 745, ^{1}H NMR 400 MHz: (DMSO-d_6, δ ppm): 2.37-2.39 (s, 3H), 7.04-7.07 (m, 2H), 7.31-7.33 (t, 1H), 7.45-7.47 (m, 4H), 7.58-7.60 (dd, 2H), 7.75-7.77 (d, 2H), 7.89-7.91 (d, 1H), 8.01-8.04 (dd, 1H), 8.23-8.25 (dd, 1H), 8.45-8.47 (s, 1H), ^{13}C NMR 400 MHz: (DMSO-d_6, δ ppm): 21.3, 98.9, 111.8, 113.0, 115.9, 117.4, 119.9, 120.1, 125.7, 126.7, 126.2, 129.3, 130.3, 130.6, 131.4, 134.4,

ISSN: 2249 –4820

Chemistry & Biology Interface, 2013, 3, 5, 314-333

139.7, 143.1, 150.0, 150.4, 152.6, 157.5, 159.4, 179.9, 183.7. Mass: [m/e (%)], M. Wt.: 464.47 anal. Calcd. for $C_{28}H_{20}N_2O_5$: C, 72.41; H, 4.34; N, 6.03; O, 17.22 Found: C, 72.40; H, 4.30; N, 6.01; O, 17.20 %.

(E)-4-hydroxy-7-methyl-3-(3-(3-(3-nitrophenyl)-1-phenyl-1H-pyrazol-4-yl)acryloyl)-2H-chromen-2-one (7n)

M.P. 238-240°C. IR (KBr) cm-1: 3645, 3545, 3128, 3043, 2996, 2858, 1734, 1620, 1527, 1440, 1356, 1300, 1228, 1143, 1103, 991, 860, 823, ^{1}H NMR 400 MHz: (DMSO-d_6, δ ppm): 2.34-2.36 (s, 3H), 7.16-7.18 (d, 1H), 7.28-7.30 (dd, 1H),7.44-7.46 (dd, 2H), 7.54-7.56 (d, 1H), 7.57-7.59 (dd, 2H), 7.63-7.65 (dd, 2H), 7.74-7.76 (dd, 1H), 8.10-8.12 (d, 2H), 8.30-8.32 (d, 2H), 8.56-8.58 (s, 1H), ^{13}C NMR 400 MHz: (DMSO-d_6, δ ppm): 21.3, 98.9, 111.8, 113.0, 117.4, 119.9, 122.7, 123.9, 125.7, 126.2, 126.7, 129.3, 130.3, 130.6, 131.4, 133.9, 139.7, 143.1, 148.4, 150.4, 152.6, 159.4, 179.9, 183.7. Mass: [m/e (%)], M. Wt.: 493.47 anal. Calcd. for $C_{28}H_{19}N_3O_6$: C, 68.15; H, 3.88; N, 8.52; O, 19.45 Found: C, 68.12; H, 3.88; N, 8.50; O, 19.42 %.

(E)-4-hydroxy-3-(3-(3-(2-hydroxyphenyl)-1-phenyl-1H-pyrazol-4-yl)acryloyl)-7-methyl-2H-chromen-2-one (7o)

M.P. 240-242°C. IR (KBr) cm^{-1}: 3622, 3115, 3057, 2885, 2831, 1710, 1616, 1510, 1442, 1461, 1372, 1296, 1234, 1101, 977, 864, ^{1}H NMR 400 MHz: (DMSO-d_6, δ ppm): 2.32-2.34 (s, 3H), 7.10-7.12 (s, 1H), 7.14-7.16 (t, 1H), 7.23-7.26 (m, 3H), 7.42-7.44 (d, 2H), 7.54-7.56 (m, 4H), 7.74-7.76 (d, 2H), 7.93-7.95 (d, 1H), 8.02-8.04 (d, 1H), 8.44-8.46 (s, 1H), ^{13}C NMR 400 MHz: (DMSO-d_6, δ ppm): 21.3, 98.9, 111.8, 113.0, 117.4, 117.8, 119.9, 120.5, 121.8, 125.7, 126.2, 129.3, 130.1, 131.4, 131.5, 139.7, 143.1, 150.0, 150.4, 155.2, 159.4, 179.9, 183.7. Mass: [m/e (%)], M. Wt.: 464.47 anal. Calcd. for $C_{28}H_{20}N_2O_5$: C, 72.41; H, 4.34; N, 6.03; O, 17.22 Found: C, 72.40; H, 4.30; N, 6.01; O, 17.20 %.

(E)-4-hydroxy-3-(3-(3-(2-methoxyphenyl)-1-phenyl-1H-pyrazol-4-yl)acryloyl)-7-methyl-2H-chromen-2-one (7p)

M.P. 246-248°C. IR (KBr) cm^{-1}: 3589, 3524, 3097, 3059, 2949, 2833, 1708, 1618, 1593, 1529, 1440, 1336, 1282, 977, 842, 750, ^{1}H NMR 400 MHz: (DMSO-d_6, δ ppm): 2.34-2.36 (s, 3H), 3.78-3.80 (s, 3H), 7.05-7.08 (m, 4H), 7.28-7.30 (t, 1H), 7.43-7.45 (m, 4H), 7.73-7.75 (d, 2H), 7.86-7.88 (m, 2H), 8.05-8.09 (dd, 1H), 8.43-8.45 (s, 1H), ^{13}C NMR 400 MHz: (DMSO-d_6, δ ppm): 21.3, 56.1, 98.9, 111.1, 111.8, 113.0, 117.4, 118.9, 119.9, 121.5, 125.7, 126.2, 129.3, 129.7, 130.3, 131.1, 131.4, 139.7, 143.1, 150.0, 150.4, 152.6, 157.3, 159.4, 179.9, 183.7. Mass: [m/e (%)], M. Wt.: 478.50 anal. Calcd. for $C_{29}H_{22}N_2O_5$: C, 72.79; H, 4.63; N, 5.85; O, 16.72 Found: C, 72.75; H, 4.60; N, 5.83; O, 16.70 %.

(E)-3-(3-(1,3-diphenyl-1H-pyrazol-4-yl)acryloyl)-4-hydroxy-7,8-dimethyl-2H-chromen-2-one (7q)

M.P. 188-190°C. IR (KBr) cm^{-1}: 3616, 3501, 3016, 2988, 2892, 1711, 1488, 1381, 1272, 1256, 1225, 997, 715, ^{1}H NMR 400 MHz: (DMSO-d_6, δ ppm): 2.07-2.09 (s, 3H), 2.33-2.35 (s, 3H), 7.04-7.06 (d, 1H), 7.10-7.12 (d, 1H), 7.42-7.44 (t, 2H), 7.51-7.53 (dd, 2H), 7.57-7.59 (dd, 2H), 7.62-7.64 (dd, 3H), 7.74-7.76 (t, 2H), 7.87-7.89 (d, 1H), 8.41-8.43 (s, 1H), ^{13}C NMR 400 MHz: (DMSO-d_6, δ ppm): 11.6, 18.8, 98.9, 113.0, 114.3, 119.9, 123.7, 124.5, 126.2, 127.5, 128.7, 128.8, 129.2, 129.3, 130.3, 131.4, 133.0, 137.9, 139.7, 150.3, 150.4, 152.6, 159.4, 179.9, 183.7. Mass: [m/e (%)], M. Wt.: 462.50 anal. Calcd. for $C_{29}H_{22}N_2O_4$: C, 75.31; H, 4.79; N, 6.06; O, 13.84 Found: C, 75.30; H, 4.78; N, 6.05; O, 13.80 %.

ISSN: 2249 –4820
Chemistry & Biology Interface, 2013, 3, 5, 314-333

(E)-4-hydroxy-7,8-dimethyl-3-(3-(1-phenyl-3-(p-tolyl)-1H-pyrazol-4-yl)acryloyl)-2H-chromen-2-one (7r)

M.P. 210-212°C. IR (KBr) cm^{-1}: 3661, 3521, 3046, 2981, 3045, 2899, 1715, 1498, 1382, 1271, 1257, 1235, 987, 732, ^{1}H NMR 400 MHz: (DMSO-d_6, δ ppm): 2.07-2.09 (s, 3H), 2.33-2.35 (s, 6H), 7.04-7.06 (d, 1H), 7.28-7.30 (t, 2H), 7.52-7.54 (dd, 2H), 7.58-7.60 (dd, 2H), 7.63-7.65 (m, 3H), 7.74-7.76 (t, 2H), 7.84-7.86 (d, 1H), 8.42-8.43 (s, 1H), ^{13}C NMR 400 MHz: (DMSO-d_6, δ ppm): 11.6, 18.8, 21.3, 98.9, 113.0, 114.3, 119.9, 123.7, 124.5, 126.2, 126.3, 128.4, 128.8, 129.3, 129.5, 130.3, 131.7, 137.9, 139.7, 150.4, 150.3, 152.6, 159.4, 179.9, 183.7. Mass: [m/e (%)], M. Wt.: 476.52 anal. Calcd. for $C_{30}H_{24}N_2O_4$: C, 75.61; H, 5.08; N, 5.88; O, 13.43 Found: C, 75.60; H, 5.07; N, 5.87; O, 13.40 %.

(E)-4-hydroxy-7,8-dimethyl-3-(3-(3-(4-nitrophenyl)-1-phenyl-1H-pyrazol-4-yl)acryloyl)-2H-chromen-2-one (7s)

M.P. 180-182°C. IR (KBr) cm^{-1}: 3620, 3547, 3086, 2998, 2857, 1712, 1616, 1498, 1384, 1284, 1219, 1097, 929, 755, 701, ^{1}H NMR 400 MHz: (DMSO-d_6, δ ppm): 2.37-2.39 (s, 3H), 2.40-2.42 (s, 3H), 7.08-7.10 (d, 1H), 7.34-7.36 (t, 1H), 7.47-7.49 (t, 2H), 7.76-7.78 (m, 3H), 7.85-7.87 (d, 2H), 8.01-8.03 (s, 1H), 8.28-8.30 (s, 1H), 8.31-8.33 (t, 2H), 8.46-8.48 (s, 1H), ^{13}C NMR 400 MHz: (DMSO-d_6, δ ppm): 11.6, 18.8, 98.9, 113.0, 114.3, 119.9, 123.7, 124.4, 124.5, 126.2, 129.3, 130.3, 131.4, 137.9, 139.1, 139.7, 147.9, 150.3, 152.6, 159.4, 179.9, 183.7. Mass: [m/e (%)], M. Wt.: 507.14 anal. Calcd. for $C_{29}H_{21}N_3O_6$: C, 68.63; H, 4.17; N, 8.28; O, 18.92 Found: C, 68.53; H, 4.12; N, 8.22; O, 18.88%.

(E)-3-(3-(3-(4-chlorophenyl)-1-phenyl-1H-pyrazol-4-yl)acryloyl)-4-hydroxy-7,8-dimethyl-2H-chromen-2-one (7t)

M.P. 204-206°C. IR (KBr) cm^{-1}: 3585, 3525, 3591, 3091, 3039, 2949, 2877, 1708, 1614, 1518, 1446, 1344, 1288, 1230, 1143, 1030, 862, 744, 701, ^{1}H NMR 400 MHz: (DMSO-d6, δ ppm): 2.07-2.09 (s, 3H), 2.33-2.35 (s, 3H), 7.02-7.04 (d, 1H), 7.26-7.28 (t, 2H), 7.52-7.54 (dd, 2H), 7.57-7.59 (dd, 2H), 7.62-7.64 (m, 3H), 7.76-7.78 (t, 2H), 7.84-7.86 (d, 1H), 8.43-8.45 (s, 1H), ^{13}C NMR 400 MHz: (DMSO-d_6, δ ppm): 11.6, 18.8, 98.9, 113.0, 114.3, 119.9, 123.7, 124.5, 126.2, 128.8, 128.9, 129.3 130.3, 131.4, 134.3, 137.9, 139.7, 150.4, 152.6, 159.4, 179.9, 183.7. Mass: [m/e (%)], M. Wt.: 496.94 anal. Calcd. for $C_{29}H_{21}ClN_2O_4$: C, 70.09; H, 4.26; Cl, 7.13; N, 5.64; O, 12.88 Found: C, 70.07; H, 4.25; Cl, 7.10; N, 5.62 %.

(E)-4-hydroxy-7,8-dimethyl-3-(3-(3-(3-nitrophenyl)-1-phenyl-1H-pyrazol-4-yl)acryloyl)-2H-chromen-2-one (7u)

M.P. 200-202°C. IR (KBr) cm^{-1}: 3645, 3134, 3076, 2974, 2875, 1718, 1612, 1591, 1525, 1433, 1350, 1296, 1220, 1095, 981, 756, ^{1}H NMR 400 MHz: (DMSO-d6, δ ppm): 2.29-2.31 (s, 3H), 2.34-2.36 (s, 3H), 7.09-7.11 (d, 1H), 7.44-7.46 (t, 1H), 7.47-7.49 (t, 2H), 7.76-7.78 (t, 1H), 7.97-7.99 (m, 2H), 8.24-8.27 (m, 2H), 8.46-8.48 (s, 1H), 8.55-8.57 (t, 1H), ^{13}C NMR 400 MHz: (DMSO-d_6, δ ppm): 11.6, 18.8, 98.9, 113.0, 114.3, 119.9, 122.7, 123.3, 123.7, 123.9, 124.5, 126.2, 128.8, 129.3, 130.6, 131.4, 133.9, 137.9, 139.7, 148.4, 150.3, 152.6, 159.4, 179.9, 183.7. M. Wt.: 507.49 anal. Calcd. for $C_{29}H_{21}N_3O_6$: C, 68.63; H, 4.17; N, 8.28; O, 18.92 Found: C, 68.60; H, 4.14; N, 8.25; O, 18.90 %.

(E)-4-hydroxy-3-(3-(3-(3-hydroxyphenyl)-1-phenyl-1H-pyrazol-4-yl)acryloyl)-7,8-dimethyl-2H-chromen-2-one (7v)

M.P. 184-186°C. IR (KBr) cm^{-1}: 3641, 3010, 2897, 2864, 1717, 1560, 1514, 1413 , 1392, 1284, 1220, 1089, 970, 833, 750, ^{1}H NMR

ISSN: 2249 –4820

Chemistry & Biology Interface, 2013, 3, 5, 314-333

400 MHz: (DMSO-d_6, δ ppm): 2.29-2.31 (s, 3H), 2.32-2.34 (s, 3H), 7.04-7.06 (d, 1H), 7.31-7.33 (t, 1H), 7.44-7.46 (m, 4H), 7.74-7.76 (t, 3H), 7.76-7.78 (d, 2H), 7.87-7.89 (d, 1H), 8.16-8.18 (d, 1H), 8.41-8.43 (s, 1H), ^{13}C NMR 400 MHz: (DMSO-d_6, δ ppm): 11.6, 18.8, 98.9, 113.0, 114.3, 115.9, 119.9, 120.1, 123.7, 124.5, 126.2, 128.8, 129.3, 130.3, 130.6, 131.4, 134.4, 137.9, 139.7, 150.4, 152.6, 157.5, 159.4, 179.9, 183.7. Mass: [m/e (%)], M. Wt.: 478.50 anal. Calcd. for $C_{29}H_{22}N_2O_5$: C, 72.79; H, 4.63; N, 5.85; O, 16.72 Found: C, 72.75; H, 4.60; N, 5.80; O, 16.70 %.

(E)-4-hydroxy-3-(3-(3-(2-hydroxyphenyl)-1-phenyl-1H-pyrazol-4-yl)acryloyl)-7,8-dimethyl-2H-chromen-2-one (7w)

M.P. 190-192°C. IR (KBr) cm^{-1}: 3564, 3107, 3043, 2947, 2889, 1712, 1614, 1595, 1529, 1450, 1294, 1240, 1093, 977, 862, 754, ^{1}H NMR 400 MHz: (DMSO-d_6, δ ppm): 2.30-2.32 (s, 3H), 2.35-2.37 (s, 3H), 7.06-7.08 (t, 1H), 7.09-7.11 (dd, 2H), 7.31-7.33 (m, 2H), 7.48-7.50 (m, 3H), 7.53-7.55 (d, 2H), 7.68-7.70 (s, 2H), 7.70-7.72 (d, 1H), 8.22-8.25 (dd, 2H), 8.46-8.48 (s, 1H), ^{13}C NMR 400 MHz: (DMSO-d_6, δ ppm): 11.6, 18.8, 98.9, 113.0, 114.3, 117.8, 119.9, 120.5, 121.8, 123.7, 124.5, 126.2, 128.8, 129.3, 130.3, 130.5, 137.9, 139.7, 150.3, 152.6, 155.2, 159.4, 179.9, 183.7. Mass: [m/e (%)], M. Wt.: 478.50 anal. Calcd. for $C_{29}H_{22}N_2O_5$: C, 72.79; H, 4.63; N, 5.85; O, 16.72 Found: C, 72.76; H, 4.60; N, 5.80; O, 16.70 %.

(E)-4-hydroxy-3-(3-(3-(2-methoxyphenyl)-1-phenyl-1H-pyrazol-4-yl)acryloyl)-7,8-dimethyl-2H-chromen-2-one (7x)

M.P. 192-194°C. IR (KBr) cm^{-1}: 3630, 3591, 3057, 2949, 2893, 1718, 1595, 1508, 1438, 1290, 1224, 1165, 1128, 1020, 977, 866, 741, ^{1}H NMR 400 MHz: (DMSO-d_6, δ ppm): 2.28-2.30 (s, 3H), 2.32-2.34 (s, 3H), 3.77-3.79 (s, 3H), 7.02-7.04 (m, 3H), 7.27-7.29 (t, 1H), 7.38-7.40 (m, 4H), 7.72-7.74 (d, 3H), 7.85-7.87 (d, 1H), 8.08-8.12 (d, 1H), 8.42-8.44 (s, 1H), ^{13}C NMR 400 MHz: (DMSO-d_6, δ ppm): 11.6, 18.8, 56.1, 98.9, 111.1, 113.0, 114.3, 118.9, 119.9, 121.5, 123.7, 126.2, 128.8, 129.3, 129.7, 130.3, 131.1, 131.4, 137.9, 139.7, 150.3, 152.6, 157.3, 159.4, 179.9, 183.7. Mass: [m/e (%)], M. Wt.: 492.52 anal. Calcd. for $C_{30}H_{24}N_2O_5$: C, 73.16; H, 4.91; N, 5.69; O, 16.24 Found: C, 73.10; H, 4.90; N, 5.65; O, 16.20 %.

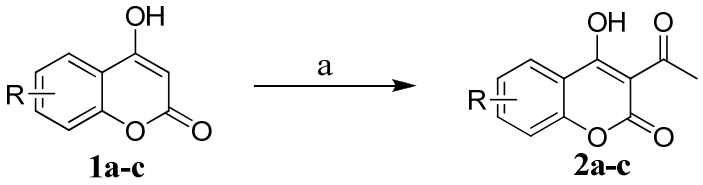
Acknowledgements

The authors are thankful to Department of Chemistry, Saurashtra University, Rajkot and specially indebted to “National Facility for Drug Discovery through New Chemical Entities (NCE's), Development & Instrumentation Support to Small Manufacturing Pharma Enterprises”, a programme under Drug & Pharma Research Support (DPRS) jointly funded by Department of Science & Technology, New Delhi, Government of Gujarat (Industries Commissionerate) & Saurashtra University, Rajkot.

ISSN: 2249 –4820

Chemistry & Biology Interface, 2013, 3, 5, 314-333

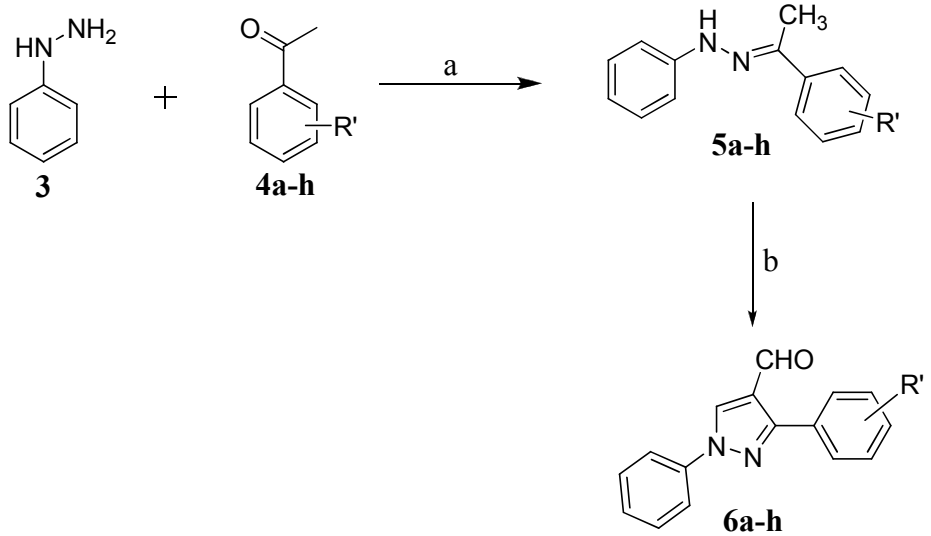
Reaction Scheme



1a-c **2a-c**

Reagents / Reaction Condition (a) : $POCl_3$, Glacial acetic acid / Reflux, 2-3 hrs.
R=6-CH_3, 7-CH_3, 7,8-diCH_3

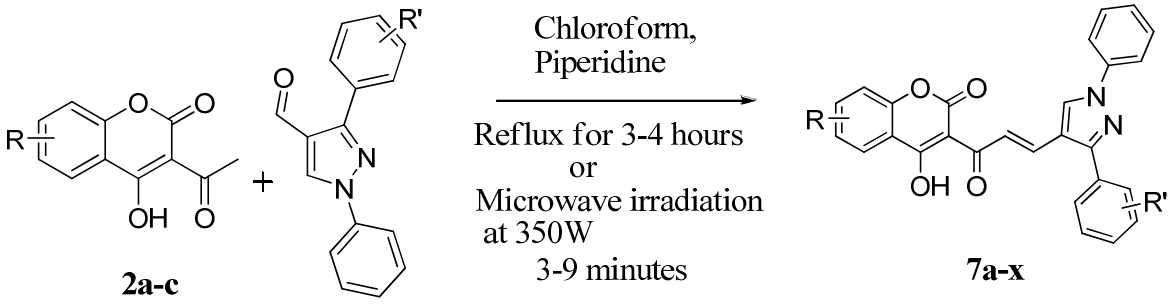
Scheme 1



3 **4a-h** **5a-h** **6a-h**

Reagents / Reaction Condition
(a) Glacial acetic acid, ethanol / reflux, 5-6 h. Where, R' = 4-H, 4-Cl, 4-F, 4-NO_2, 3-NO_2 ,etc.
(b) DMF – $POCl_3$ / 70-80°C, 5-6 h. Where, R' = 4-H, 4-Cl, 4-F, 4-NO_2, 3-NO_2, etc.

Scheme 2



2a-c **6a-h** **7a-x**

Scheme 3

ISSN: 2249 –4820

Chemistry & Biology Interface, 2013, 3, 5, 314-333

Table 1. Optimization of yield for the microwave assisted synthesis of **7a, 7i** and **7q** using different solvents.

Entry as	Solvent	Time[a] (min)	Yield[b] %
7a	Chloroform	7	85
7i	Chloroform	5	79
7q	Chloroform	7	80
7a	Dichloromethane	12	70
7i	Dichloromethane	9	76
7q	Dichloromethane	10	68
7a	DMF	12	62
7i	DMF	15	70
7q	DMF	10	66
7a	Ethyl Acetate	10	64
7i	Ethyl Acetate	11	68
7q	Ethyl Acetate	14	70

[a]Time taken for the synthesis by microwave assisted method.
[b]Isolated yields after purification by microwave assisted method.

Table 2. Synthesis of highly functionalized chalcone derivatives by microwave assisted method of synthesis.

Entry	Substitution		Time[a] (minutes)	Melting Point (^{0}C)
	R	R'		
7a	6-CH_3	H	7	220-222
7b	6-CH_3	4-CH_3	8	230-232
7c	6-CH_3	4-NO_2	3	224-226
7d	6-CH_3	4-Cl	8	228-230
7e	6-CH_3	3-OH	6	232-234
7f	6-CH_3	3-NO_2	8	218-220
7g	6-CH_3	2-OH	5	172-174
7h	6-CH_3	2-OCH_3	9	212-214
7i	7-CH_3	H	5	180-182
7j	7-CH_3	4-CH_3	5	190-192
7k	7-CH_3	4-NO_2	7	200-202
7l	7-CH_3	4-Cl	5	184-186
7m	7-CH_3	3-OH	8	192-194
7n	7-CH_3	3-NO_2	9	188-190
7o	7-CH_3	2-OH	7	210-212
7p	7-CH_3	2-OCH_3	8	204-206
7q	7,8-di CH_3	H	7	234-236
7r	7,8-di CH_3	4-CH_3	3	240-242
7s	7,8-di CH_3	4-NO_2	7	246-248
7t	7,8-di CH_3	4-Cl	8	238-240
7u	7,8-di CH_3	3-NO_2	7	244-246

ISSN: 2249 –4820

Chemistry & Biology Interface, 2013, 3, 5, 314-333

7v	7,8-di CH_3	3-OH	8	242-244
7w	7,8-di CH_3	2-OH	6	248-250
7x	7,8-di CH_3	2-OCH_3	9	232-234

[a]Time taken for the synthesis by microwave assisted method

Table 3. Comparison of yield (%) of the chalcone derivatives obtained using microwave assisted as well as conventional method of synthesis.

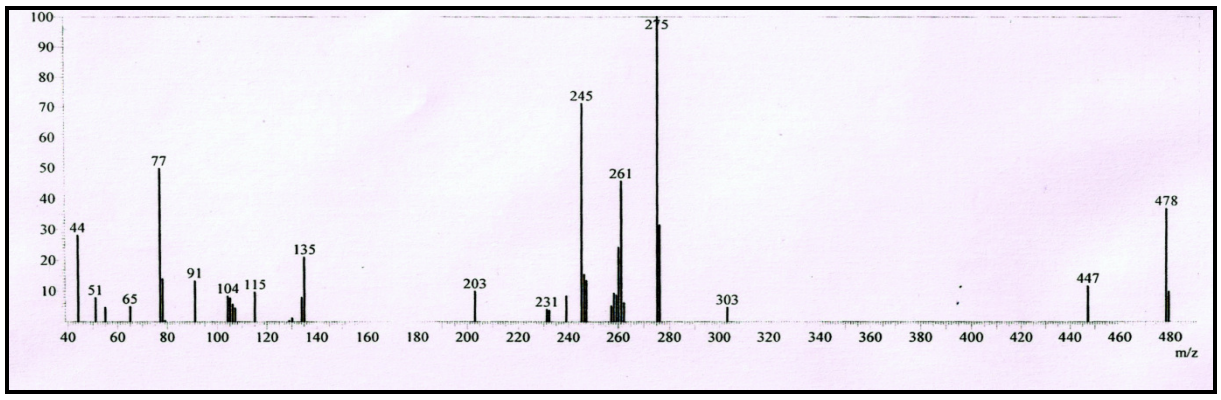
Compound	Yield (%) obtained by the method of synthesis	
	Microwave assisted method	Conventional method
7a	85	66
7b	82	67
7c	83	58
7d	82	62
7e	84	66
7f	84	63
7g	78	58
7h	70	48
7i	79	63
7j	77	54
7k	70	56
7l	72	62
7m	76	68
7n	83	60
7o	69	62
7p	73	46
7q	80	58
7r	79	55
7s	83	57
7t	83	63
7u	80	54
7v	84	58
7w	78	52
7x	76	49

ISSN: 2249 –4820

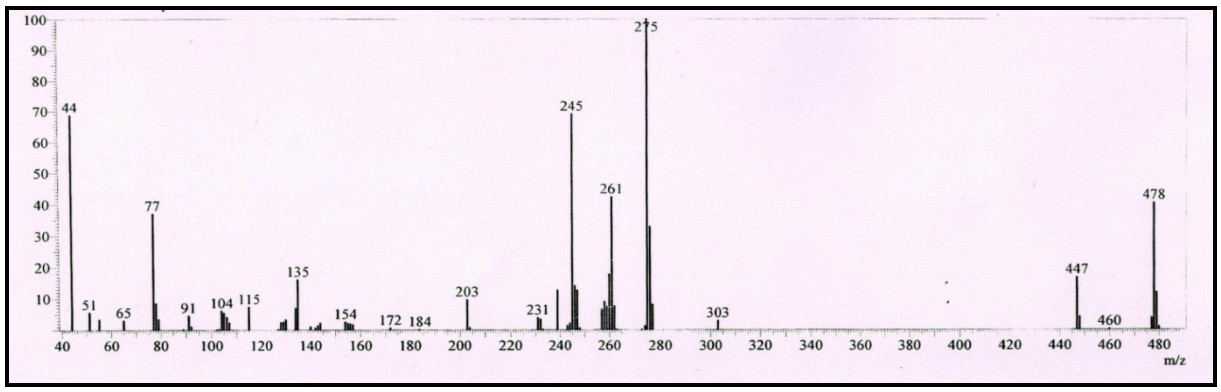
Chemistry & Biology Interface, 2013, 3, 5, 314-333

Supporting Data:

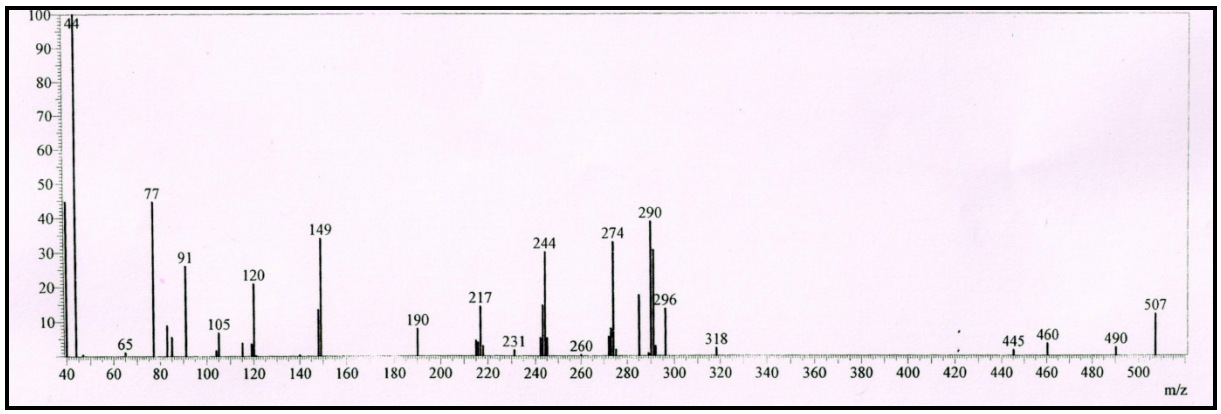
Mass spectra of 7h:



Mass spectra of 7p:



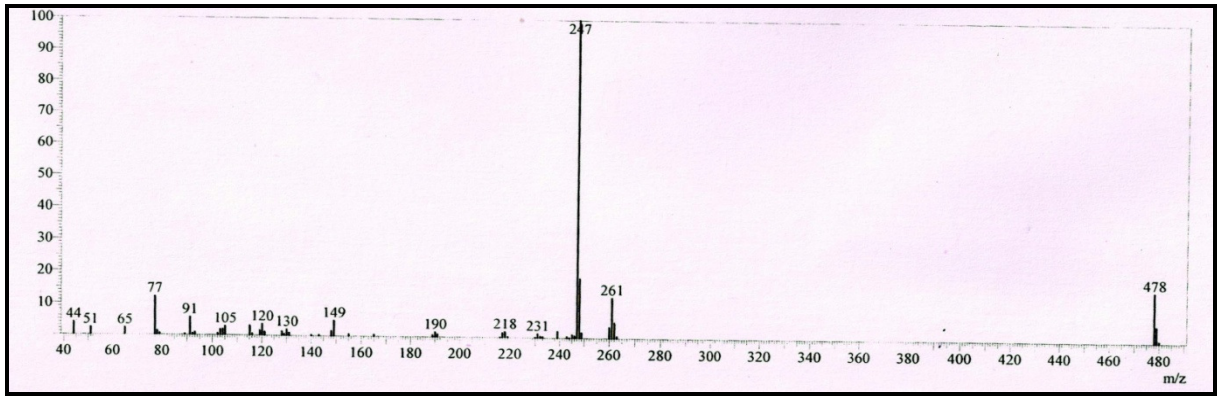
Mass spectra of 7u:



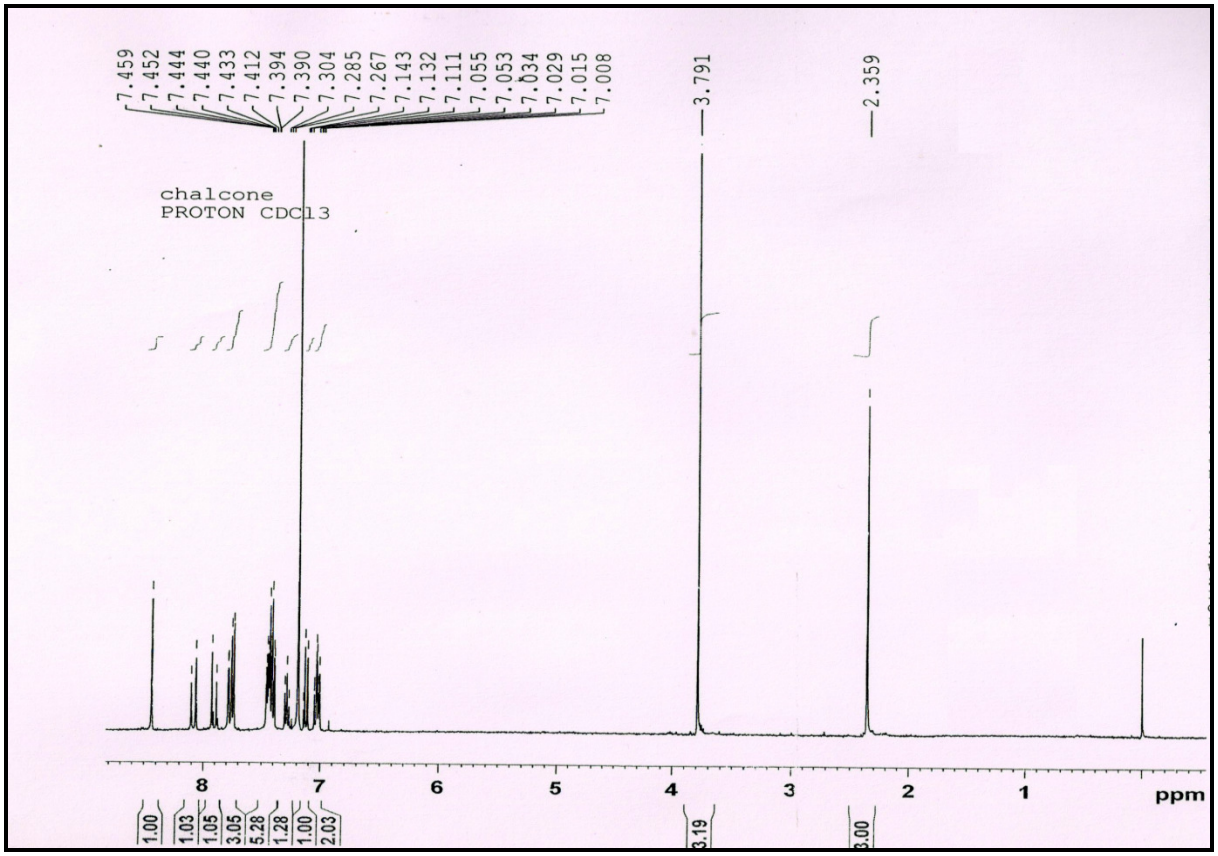
ISSN: 2249 –4820

Chemistry & Biology Interface, 2013, 3, 5, 314-333

Mass spectra of 7w:



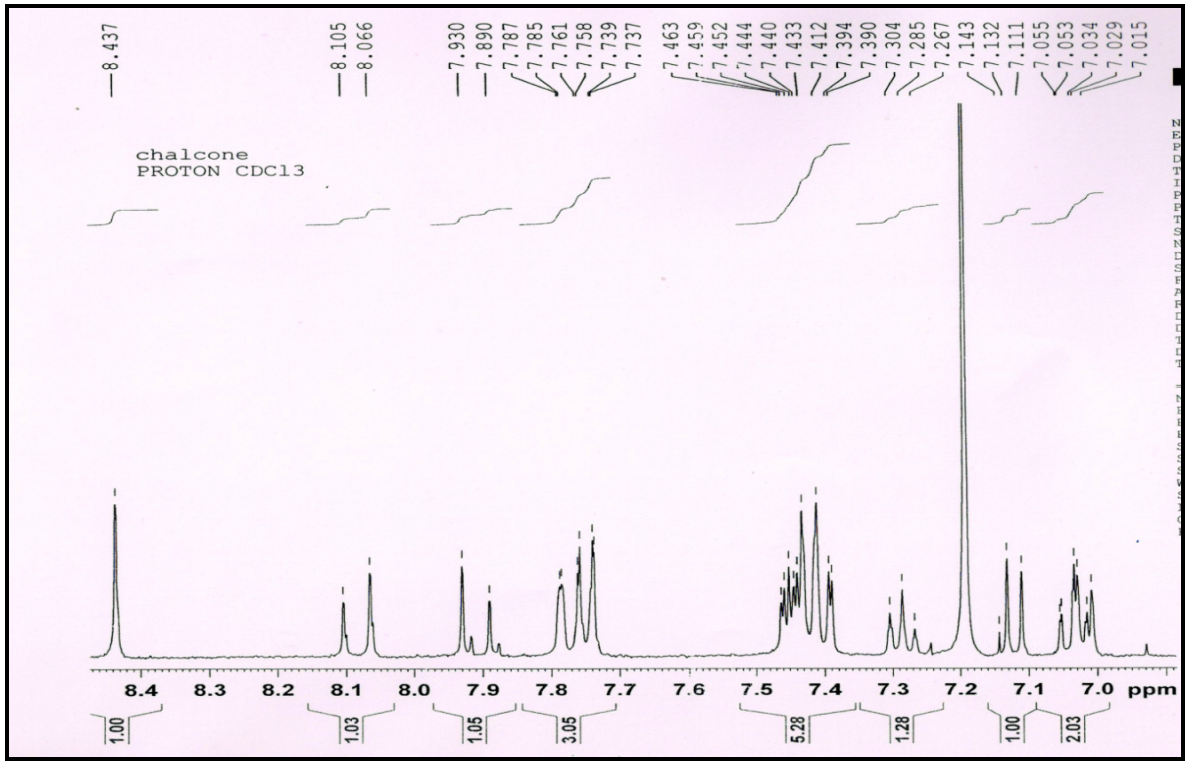
^{1}H NMR spectra of 7h:



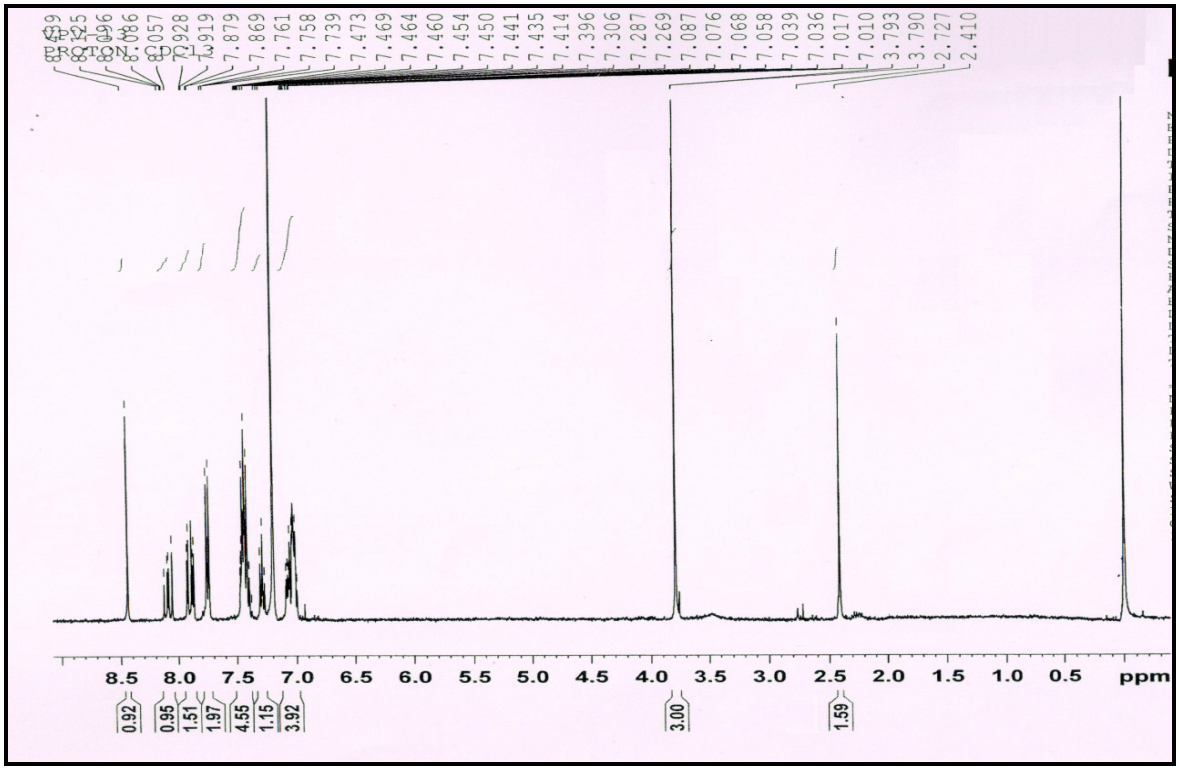
ISSN: 2249 –4820

Chemistry & Biology Interface, 2013, 3, 5, 314-333

^{1}H NMR spectra of 7h (expanded):



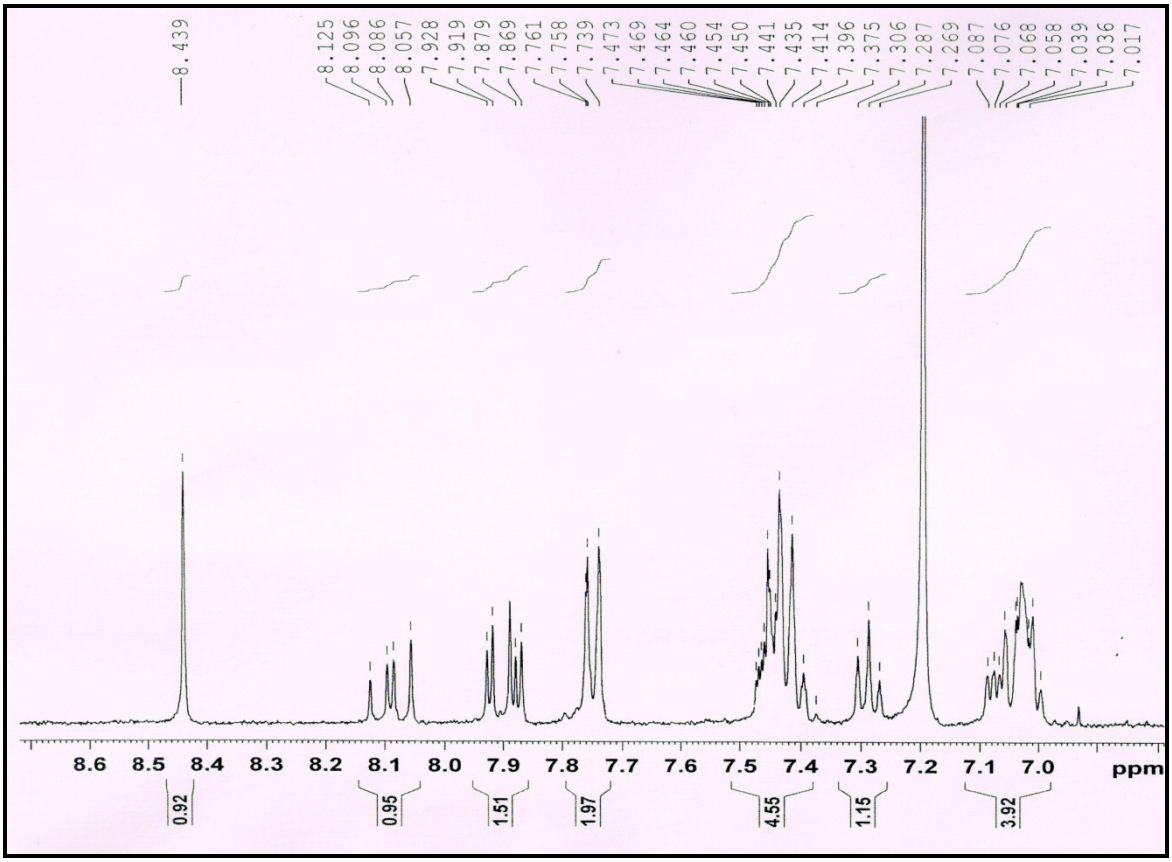
^{1}H NMR spectra of 7p:



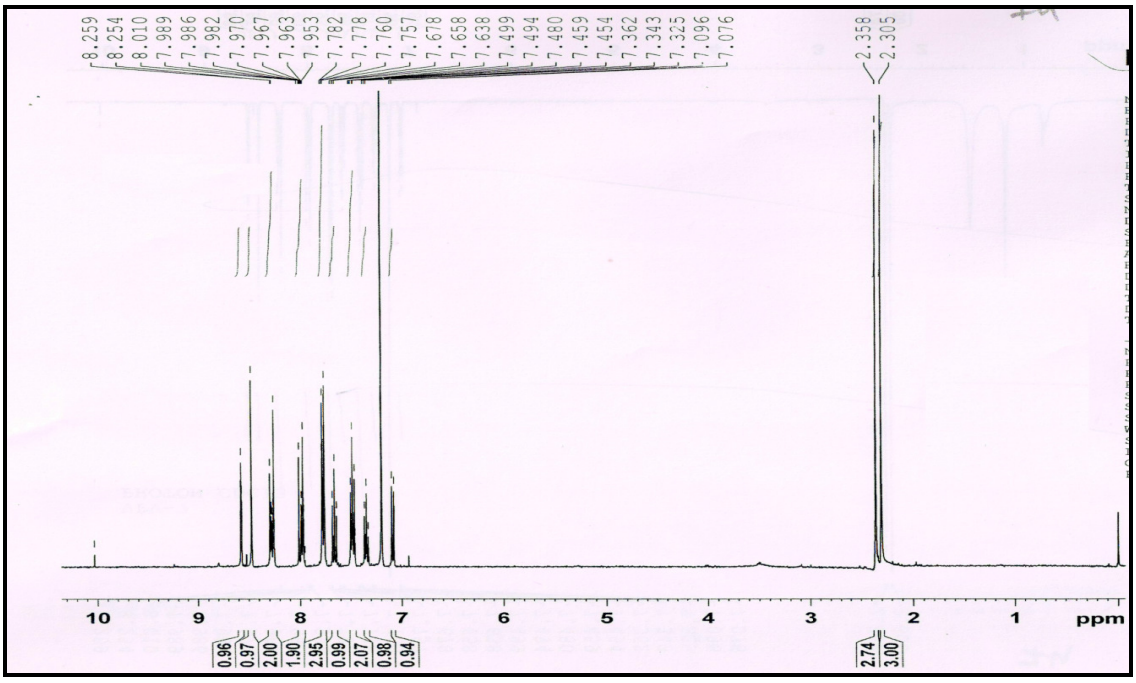
ISSN: 2249 –4820

Chemistry & Biology Interface, 2013, 3, 5, 314-333

^{1}H NMR spectra of 7p: (expanded)



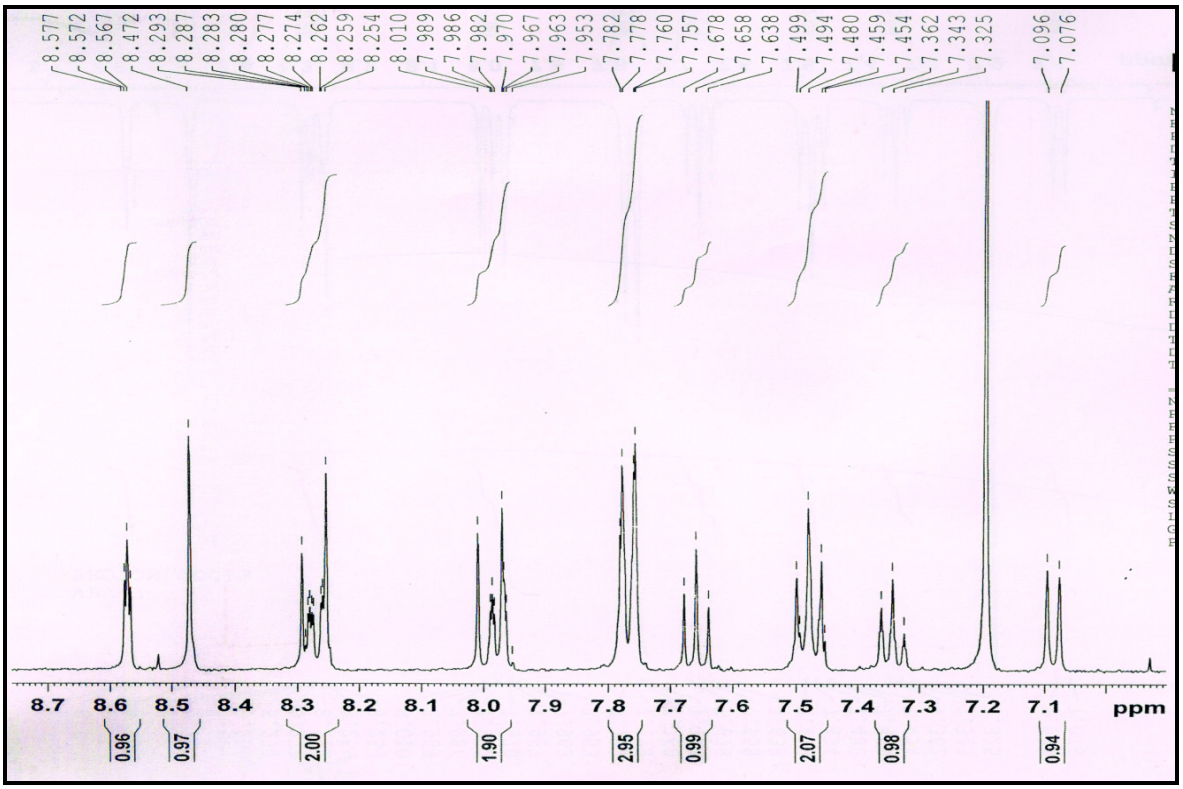
^{1}H NMR spectra of 7u:



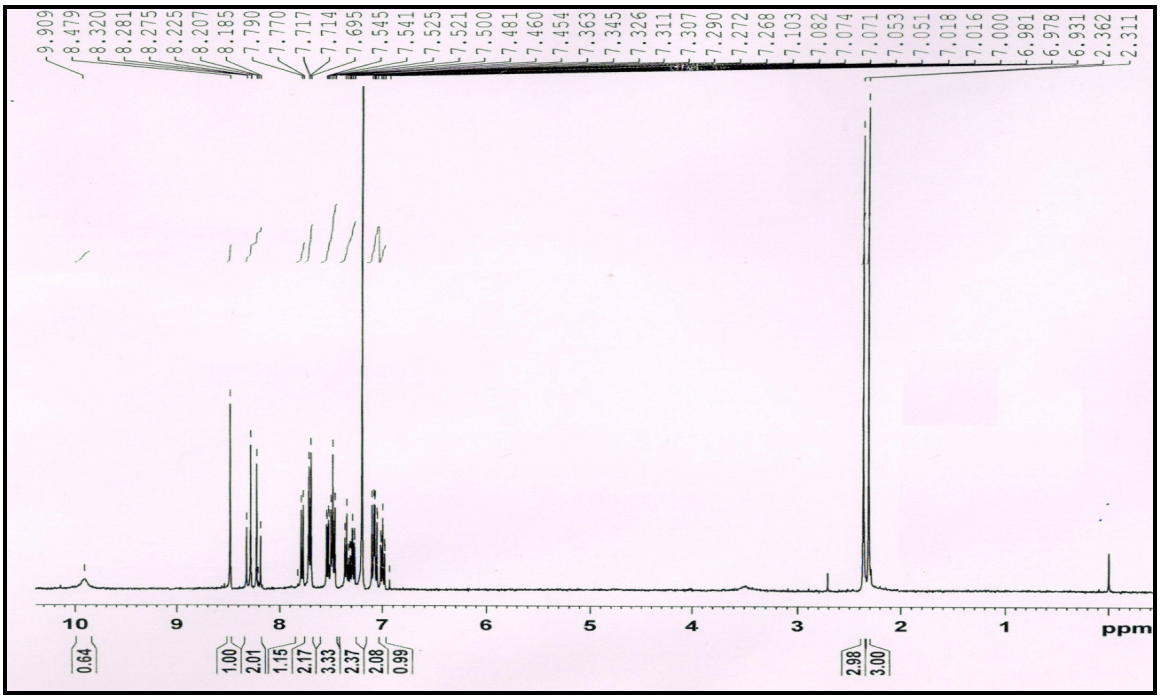
ISSN: 2249 –4820

Chemistry & Biology Interface, 2013, 3, 5, 314-333

^{1}HNMR spectra of 7u (expanded):



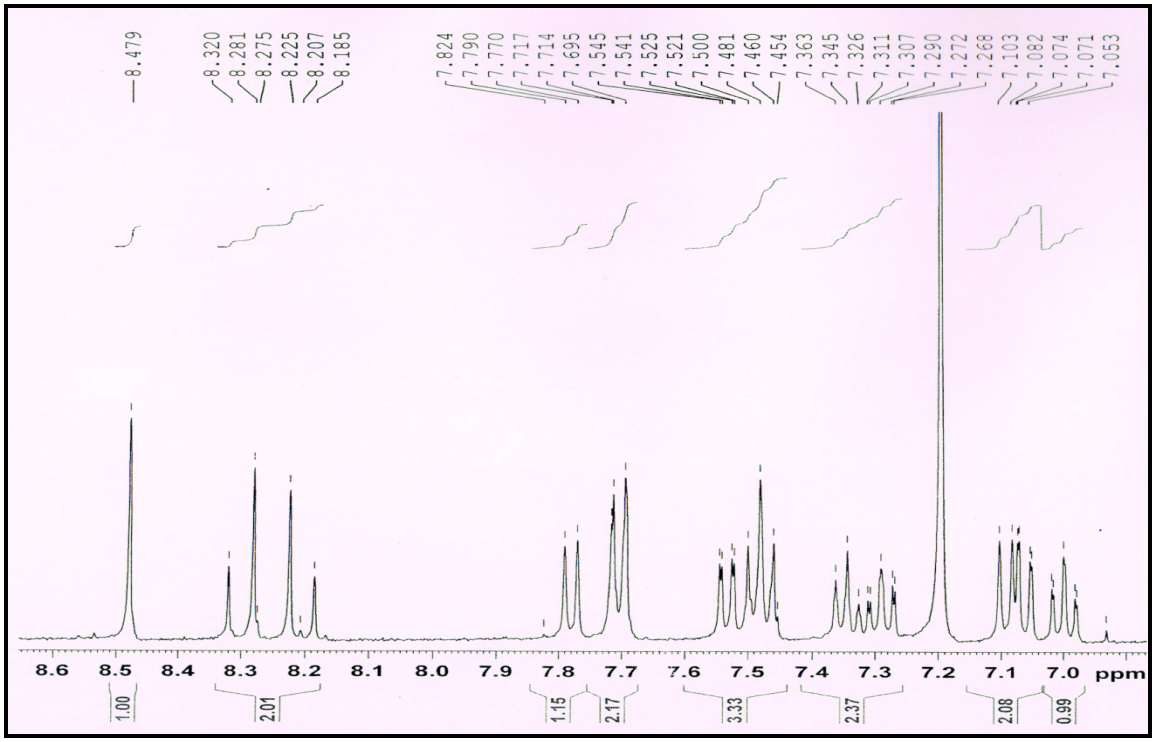
^{1}H NMR spectra of 7w:



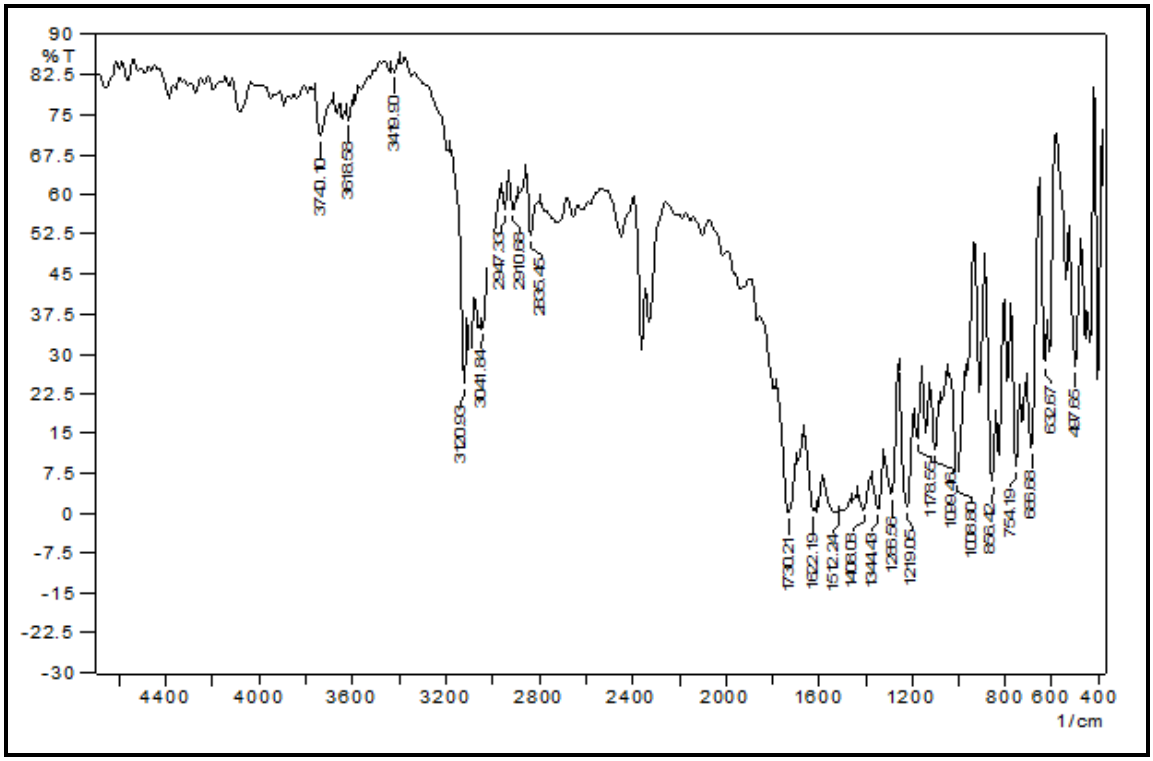
ISSN: 2249 –4820

Chemistry & Biology Interface, 2013, 3, 5, 314-333

^{1}HNMR spectra of 7w (expanded):



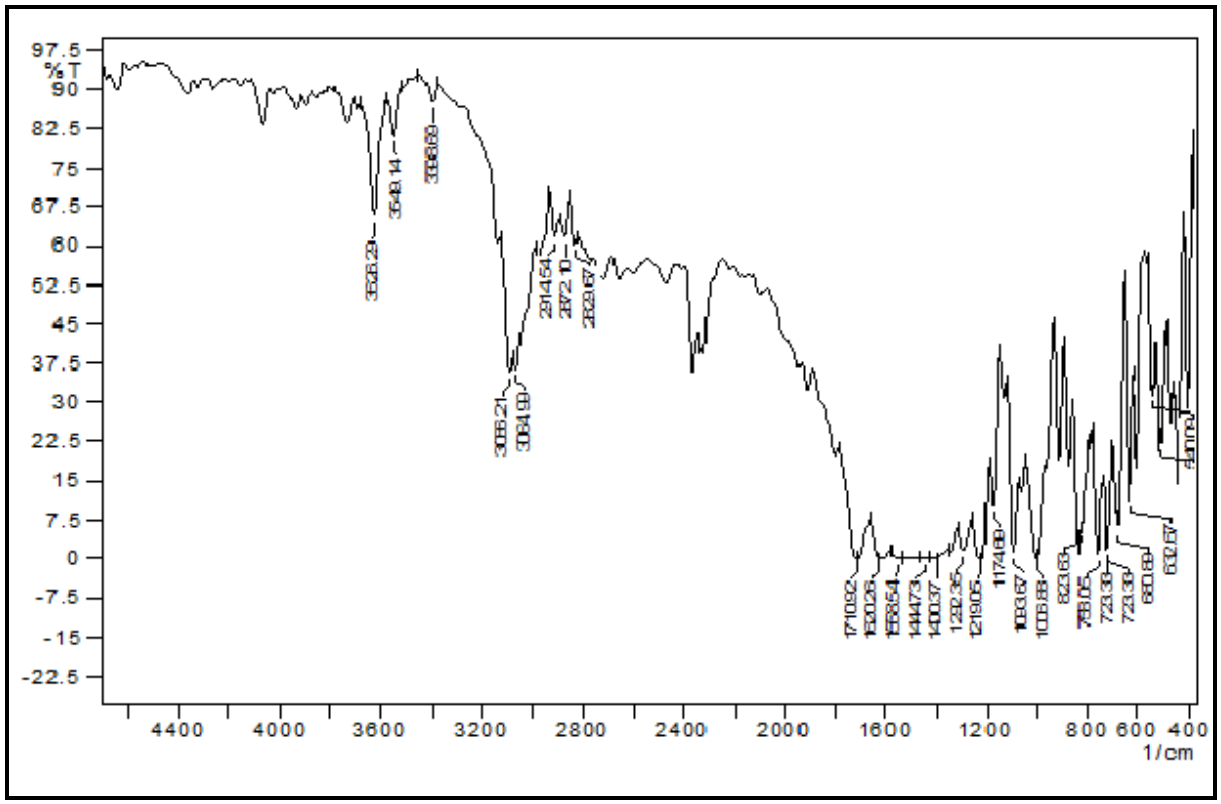
IR spectra of 7c:



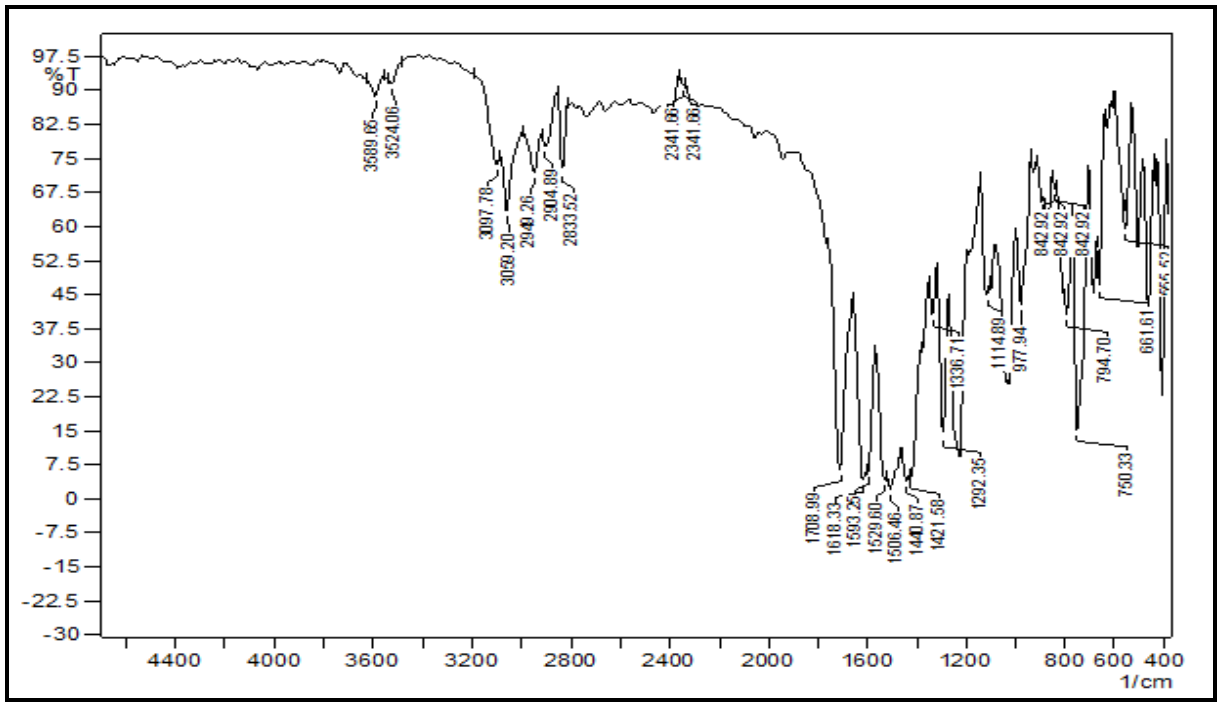
ISSN: 2249 –4820

Chemistry & Biology Interface, 2013, 3, 5, 314-333

IR spectra of 7e:



IR spectra of 7p:



ISSN: 2249 –4820

Chemistry & Biology Interface, 2013, 3, 5, 314-333

References

[1] R. D. H. Murray, J. Mendez, S. A. Brown; The Natural Coumarins: Occurrence, Chemistry and Biochemistry, Wiley & Sons, New York, NY, USA, **1982**.

[2] R. O'Kennedy, R.D. Thornes; Coumarins: Biology, Applications and Mode of Action, Wiley & Sons, Chichester, UK, **1997**.

[3] K. C. Fylaktakidou, D. J. Hadjipavlou-Litina, K. E. Litinas, D. N. Nicolaides, "Natural and Synthetic Coumarin Derivatives with Anti-inflammatory/Antioxidant Activity", Curr. Pharm. Des., **2004**, 10, 3813-3833.

[4] L. Santana, E. Uriarte, L. Dalla Via, O. Gia, "A New Benzoangelicin with Strong Photobiological Activity", Bioorg. Med. Chem. Lett., **2000**, 10, 135-137.

[5] A. Guiotto, A. Chilin, P. Manzini, F. Dall'Acqua, F. Bordin, P. Rodighiero, "Synthesis and Antiproliferative Activity of Furocoumarin Isosters", Farmaco, **1995**, 50, 479-488.

[6] P. Ploypradith, C. Mahidol, P. Sahakitpichan, S. Wongbundit, S. Ruchirawat, "A Highly Efficient Synthesis of Lamellarins K And L by The Michael Addition Ring-Closure Reaction of Benzyldihydro Isoquinoline Derivatives with Ethoxycarbonyl-B-Nitrostyrenes", Angew. Chem. Int. Ed., **2004**, 43, 866-868.

[7] M. A. Musa, J. S. Cooperwood, M. O. Khan, Curr. Med. Chem., **2008**, 15, 2664-2679.

[8] Y. Dong, Q. Shi, Y. N. Liu, X. Wang, K. F. Bastow, K. H. Lee, J. Med. Chem., **2009,** 52, 3586-3590.

[9] K. V. Sashidhara, S. R. Avula, K. Sharma, G. R. Palnati, S. R. Bathula, European Journal of Medicinal Chemistry, **2013**, 60, 120-127.

[10] W. Barrow, W. Suling, L.Westbrook, E. Borrow, Y. Lin, M. Flavin, "Anti-HIV Natural Product (+)-Calanolide A is Active Against Both Drug-Susceptible and Drug-Resistant Strains of Mycobacterium Tuberculosis", Bioorg. Med. Chem., **2004**, 12, 1199-1207.

[11] F. Uchiumi, T. Hatano, H. Ito, T. Yoshida, S. Tanuma, "Transcriptional Suppression of the HIV Promoter by Natural Compounds", Antiviral Res., **2003**, 58, 89-98.

[12] N. Hamdi, M. Saoud, A. Romerosa, "Synthesis, Spectroscopic and Antibacterial Investigations of New Hydroxyl Ethers and Heterocyclic Coumarin Derivatives", J. Heterocycl. Chem., **2008**, 45, 1835-1842.

[13] N. Hamdi, C. Lidrissi, M. Saoud, A. Romerosa, H. Zarrouk, "Synthesis of Some New Biologically Active Coumarin Derivatives", Chem. Heterocycl. Comp., **2006**, 42, 320-325.

[14] K. Venkateshwara Rao and V. Sundaramurthy, "A convenient one-step synthesis of 3-acetyl-4-hydroxycoumarins", Proc. Indian. Acad. Sci., **1975**, 81(3), 118-123.

[15] L. F. Tietze, A. Steinmetz, F. Balkenhohl, Bioorg. Med. Chem. Lett., **1997**, 7, 1303.

[16] J. Regan, S. Breitfelder, et al., J. Med. Chem., **2002**, 45, 2994.

[17] H. G. Garg, A. Singhal, J. M. L. J. Mathur, Pharm. Sci., **1973**, 62, 494.

[18] R. G. Patel, C. K. Patel, V. I. Patel, D. J. Sen, B. Panigrahi, C. N. Patel, "Synthesis and biological evaluation of anti-tubercular activity of some synthesized pyrazole derivatives", Journal of Chemical and Pharmaceutical Research, **2010**, 2(2), 112-117.

[19] V. Vinayaditya, A. Argal, V. Mishra, R. Raghuvanshi, S. Agnihotri, "Synthesis, structural analysis & biological evaluation of anticonvulsant activity of pyrazole derivatives containing thiourea", International Journal of Research in Pharmacy and Science, **2011**, 1(3), 125-146.

[20] P. N. Balaji, L. K. Lakshmi, K. Mohan, K. Revathi, A. Chamundeswari, P. M. Indrani, "In-vitro anti-inflammatory and antimicrobial activity of synthesized some novel pyrazole derivatives from coumarin chalcones", Pharmacia Sinica, **2012**, 3(6), 685-689.

[21] K. Divekar, S. Shivakumar, N. Kavitha, V. Murugan, M. Devgun, "Synthesis and evaluation of some new pyrazole derivatives as antimicrobial agents", Research Journal of Pharmacy and Technology, **2010**, 3(4), 1039-1043.

[22] K. L. Kees, J. J. Jr. Fitzgerald, K. E. Steiner, J. F. Mattes, B. Mihan, T. Tosi, D. Mondoro, M. L. Mccaleb, J. Med. Chem., **1996**, 39, 3920.

[23] Z. Nowakowska, Eur. J. Med. Chem., **2007**, 42, 125.

[24] A. Modzelewska, C. Pettit, G. Achanta, et al., Bioorg. Med. Chem., **2006**, 14, 3491.

[25] A. A. Valla, B. Valla, D. Cartier, et al. Eur. J. Med. Chem., **2006**, 41, 142.

[26] P. Boeck, C. A. B. Falca, P. C. Leal, et al. Bioorg. Med. Chem., **2006**, 14, 1538.

[27] S. H. Lee, G. S. Seo, J. Y. Kim, et al. Eur. J. Pharmacol., **2006**, 178-532.

[28] S. Cheenpracha, C. Karalai, C. Ponglimanont, et al. Bioorg. Med. Chem., **2006**, 14, 1710.

[29] O. Nerya, R. Musa, S. Khatib, et al. Phytochemistry, **2004**, 65, 1384.

[30] M. Chen, L. Zhai, S. B. Christensen, et al. Antimicrob. Agents Chemother, **2001**, 45, 2023.

[31] (a) M. J. Graneto, R. G. Kurumbail, M. L.Vazquez, H. S. Shieh, J. L. Pawlitz, J. M. Williams, W. C. Stallings, L. Geng, A. S. Naraian, F. J. Koszyk, M. A. Stealey, X. D. Xu, R. M. Weier, G. J. Hanson, R. J. Mourey, R. P.Compton, S. J. Mnich, G. D. Anderson, J. B. Monahan, R. J. Devraj, Med. Chem. **2007**, 50, 5712. (b) S. Kuettel, A. Zambon, M. Kaiser, R. Brun, L. Scapozza, R. J. Perozzo, Med. Chem. **2007**, 50, 5833.

[32] S. Thakrar, N. Pandya, H. Vala, A. Bavishi, A. Radadiya, C. Pannecouque and A. K. Shah, Synthesis and anti-HIV activity of novel N-((1, 3-substituted diphenyl-1H-pyrazole-4-yl) methylene)-2-methylindoline-1-amine derivatives using MTT method, Chemistry & Biology Interface, **2012**, 2, 2, 107-113.

[33] S.Thakrar, A. K. Shah, A rapid and highly efficient microwave synthesis of highly functionalized chalcones derivatives, International Journal of ChemTech Research, **2012**, 4(1).