MAY 2014 – JULY 2014, Vol. 4, No. 3; 1888-1893. E- ISSN: 2249 –1929

Journal of Chemical, Biological and Physical Sciences

An International Peer Review E-3 Journal of Sciences

*Available online at*www.jcbsc.org

Section A: Chemical Sciences

CODEN (USA): JCBPAT Research Article

Microwave Assisted synthesis of 1(2-hydroxy phenyl) -3(1, 3-diphenyl-1*H*- pyrazol-4-yl) -2 (aroyl) -2- propen -1-ones

S.B. Pawar*

P.G. Department of Chemistry, Shri Anand College Pathardi, Ahmednagar. Maharashtra, India

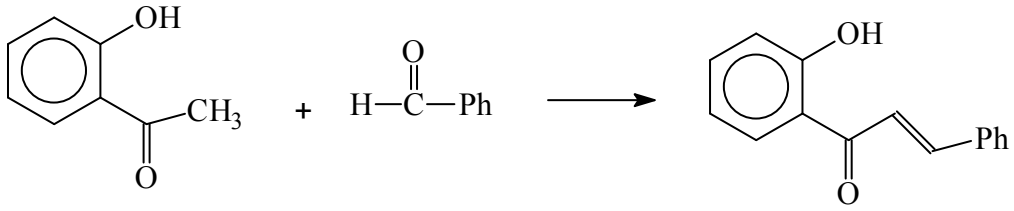
Received: 22 April 2014**; Revised:** 14 May 2014**; Accepted:** 25 May 2014

Abstract: The 1(2-hydroxy phenyl),3-(1,3-diphenyl-1*H*-pyrazol-4-yl)2-(aroyl) 2-propen -1-ones have been synthesized by condensation of 2-hydroxy 1,3 diketone and 1,3 diphenyl -1*H*-pyrazol-4-carboxaldehyde by Microwave irradiation method

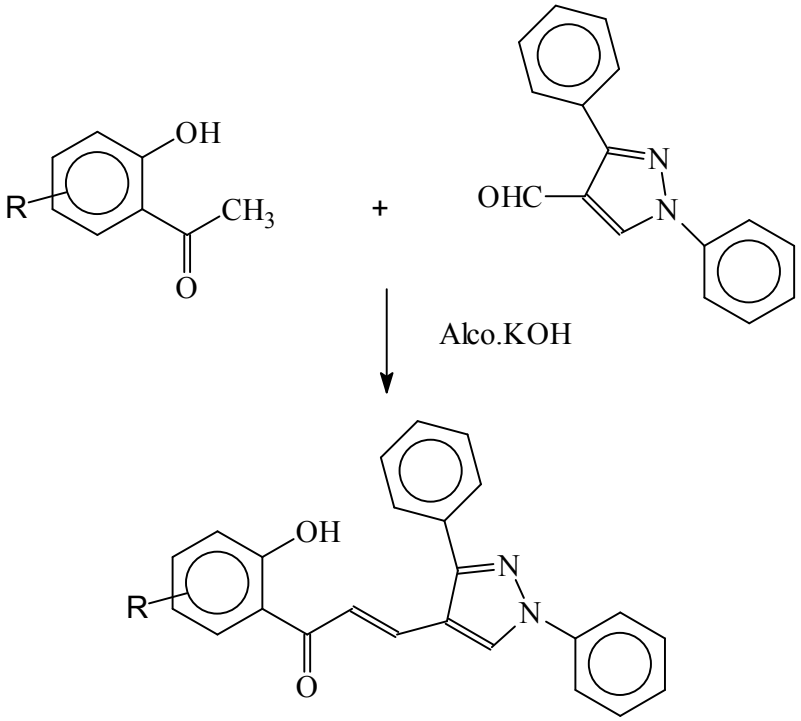
Keywords: Synthesis, Carboxaldehyde, β-Diketones, Microwave.

INTRODUCTION

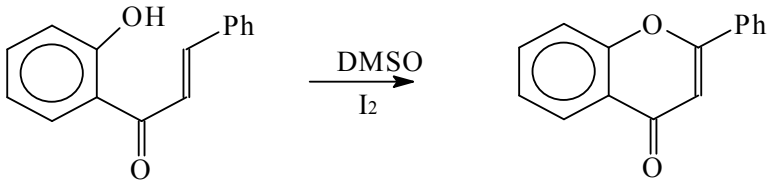
The chemistry of chalcone has assumed importance because of their versatility as an effective synthon in the synthesis of many organic compounds further they are also associated with wide spectrum of pharmacological activities. Synthesis of chalcone from o. hydroxy acetophenone & aromatic aldehyde is well known.



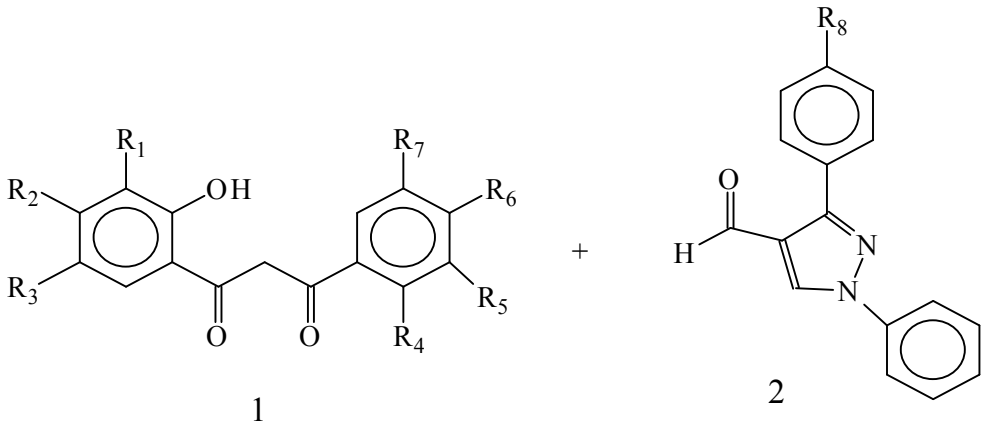
B.K.Karale and M.S.Shingare reported preparation of chalcones[1] containing pyrazole moiety having biological activity.



Majoli & kelin reported the synthetic applications of 1, 3 diketone for synthesis of carbocycles & their modern application in enantioselectivity, combinational solid phase & multi step organic synthesis[2]. 1, 3, Diketones are used for the preparation of pyrazoles[3-5].El-Ansary has studied the synthesis & analgesic properties of some 1,3 diketones[6]. 1, 3 Dicarbonyl compounds are also used in the Biginelli reaction for the preparation of 5-acyl 1,2,3,4-tetrahydro-pyrimidine-2-thiones[7-9]. 1,3 Diketones[10-11] are also used for preparation of pyrimidine derivatives. Chalcones are used to prepare chromones by cyclisation in DMSO&iodine[12-14].



Iqbal & *et al* have reported a one-step synthesis of 3-iodo flavones by reaction of 2-hydroxy chalcone with I_2 / sulphuric acid / DMSO system[15].Kushawaha[16] and coworkers have reported antibacterial and antifungal activities in substituted 3-(2-furyl) acylophenones. Activities[17-20] have been possesed by acrylophenones having pyridyl, quinolyl and furyl substituents. Some pyridyl, thienyl & furyl substituted acrylophenones have claimed bacteriostatic & tuberculostatic activities.Agasimudin[21] have synthesized number of acrylophenones and reported the importance of halogen & hydroxyl substituents in benzenoid part to enthance the bacteriostatic activity.Chalcones are versatile synthons and can be converted into a large number of heterocyclic compounds having pharmacological & biological importance.



Present Work: In the present work 1(2-hydroxy phenyl),3-(1,3-diphenyl-1*H*-pyrazol-4-yl)2-(aroyl) 2-propen -1-ones **3** have been synthesized by condensation of 2-hydroxy 1,3 diketone **1** and 1,3 diphenyl -1*H*-pyrazol-4-carboxaldehyde **2** by Microwave irradiation method .

EXPERIMENTAL

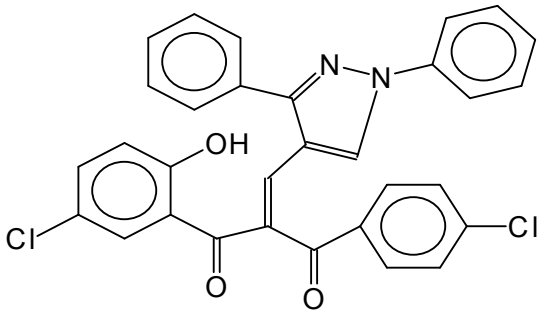
General procedure for synthesis of 1(2-hydroxy phenyl)-3(1,3-diphenyl-1*H*- pyrazol-4-yl) -2 (aroyl) -2-propen -1-ones A mixture of **1** (0.001 mole) and **2** (0.001 mole) was dissolved in absolute ethyl alcohol (40 ml), to this reaction mixture five drops of piperidine were added. The reaction mixture was kept in microwave oven of model Electrolux at 300 powers for 8-10 minutes. The reaction is monitored by TLC. Then reaction mixture was cooled. During cooling, crystal starts to develop in the reaction flask. Reaction mixture kept for overnight. The product obtained was recrystallized by acetic acid. The compounds synthesized by above method are listed in table with their physical constants and percentage yield. Their structures have been confirmed by I.R. and NMR spectra.

Table

Sr. No	R1	R2	R3	R4	R5	R6	R7	R8	M.P.^{0}C	time In Min.	% Yield
3a.	H	CH_3	Cl	H	H	H	H	H	228	8	62
3b	H	H	Cl	H	H	Cl	H	H	195	8	68
3c.	CH_3	H	CH_3	H	OCH_3	H	OCH_3	H	198	10	65
3d	H	H	Br	Cl	H	Cl	H	NO_2	285	9	48
3e.	H	CH_3	Cl	H	H	Cl	H	H	200	8	68
3f	H	H	Cl	H	H	H	H	H	205	10	56
3g.	H	H	Cl	Cl	H	H	H	H	197	10	58
3h	CH_3	H	Cl	H	OCH_3	H	OCH_3	H	205	8	57
3i.	H	H	Cl	H	H	Cl	H	CH_3	197	8	61
3j.	H	H	CH_3	H	H	OCH_3	OCH_3	CH_3	276	9	42
3k	H	H	Cl	H	H	H	H	CH_3	215	8	59
3l	CH_3	H	CH_3	H	OCH_3	H	OCH_3	NO_2	250	9	53

RESULTS & DISCUSSION

In present investigation The 1(2-hydroxy phenyl),3-(1,3-diphenyl-1*H*-pyrazol-4-yl)2-(aroyl) 2-propen -1-ones have been synthesized by condensation of 2-hydroxy 1,3 diketone and 1,3 diphenyl -1*H*-pyrazol-4-carboxaldehyde by Microwave irradiation method. I.R. spectra of representative compound of this series were scanned by using Perkin Elmer FT spectrophotometer. It showed following characteristic absorption bands in cm^{-1}. The NMR spectra of representative compound of this series were scanned on Varian 300 MHz spectrophotometer in DMSO as solvent and TMS is used as an internal standard. Peak values are shown in δ ppm.



3b

IR spectrum (KBr disc) of representative compound (3b) showed following characteristic absorption bands

- 3449 cm^{-1} due to O-H stretching
- 1663 cm^{-1} due to C=O stretching
- 1616 cm^{-1} due to C=O stretching
- 1584 cm^{-1} due to C=N stretching

NMR Spectra: The NMR spectra of representative compounds in this series were scanned on Varian 300 MHz spectrophotometer using DMSO-d6 and $CDCl_3$ as solvent and TMS used as an internal standard. The compound 3b showed following characteristic signals in δ ppm.

- 7.9 1H singlet OH
- 7.79 1H singlet pyrazole H
- 6.00 1H singlet =C-H
- 6.7-7.7 17H multiplet Ar-H

Table

Sr. No	R1	R2	R3	R4	R5	R6	R7	R8	M.P.^{0}C	time In Min.	% Yield
3a.	H	CH_3	Cl	H	H	H	H	H	228	8	62
3b	H	H	Cl	H	H	Cl	H	H	195	8	68
3c.	CH_3	H	CH_3	H	OCH_3	H	OCH_3	H	198	10	65
3d	H	H	Br	Cl	H	Cl	H	NO_2	285	9	48
3e.	H	CH_3	Cl	H	H	Cl	H	H	200	8	68
3f	H	H	Cl	H	H	H	H	H	205	10	56
3g.	H	H	Cl	Cl	H	H	H	H	197	10	58
3h	CH_3	H	Cl	H	OCH_3	H	OCH_3	H	205	8	57
3i.	H	H	Cl	H	H	Cl	H	CH_3	197	8	61
3j.	H	H	CH_3	H	H	OCH_3	OCH_3	CH_3	276	9	42
3k	H	H	Cl	H	H	H	H	CH_3	215	8	59
3l	CH_3	H	CH_3	H	OCH_3	H	OCH_3	NO_2	250	9	53

REFERENCES

1. B.K. Karale, V.P. Chavan, A.S. Mane, C.H. Gill and M.S. Shingare. *The Korean Chem. Soc*. 2000, **10**(2) 84.
2. A.V. Kel'in and Majjoli. *Current Organic Chemistry*.2003, **7**, 1855.
3. S. Nigam, Y.C. Joshi and P. Joshi. *Heteroclyclic communication*.2003**, 9**(4), 405.
4. G. Varvounis Flamegos and G. Pilidis. *Adv.Heterocyclic Chem*. 2001, **80**, 72.
5. B. Stanovnik and J. Steve. *Pyrazoles in Sci of Synthesis*, Houben Weyl. *"Methods of organic transformations*". George Thieme Verlag Stuttgart. 2002, **12**. 15-225.
6. A.K. El Ansary, Gypt. *J. Pharm. Sci.* 1991, **32**,769.
7. K.S. Atwal, B.N. Swanson, E. Ungar, D.M. Floyd, S.A. Hedberg, B.C.O'Reilly, J.E.T. Corrie. *J.Med. Chem*. 1991, **34**, 80.
8. C.O. Kappe. *Tetraledron*. 1993, **49**, 6937.
9. P. Biginelli. Gazz. Chim. *et al*. 1993, **23**,360.
10. A.K. Panday, M. Bardhan and C.S. Panda. *Indian J. of Chem.* 2003, **42**B. 910.
11. U. Ghosh, J.A. Katzenellenbogen. *J. Heterocyclic Chem.* 2002, **39**, 1101.
12. J.A.S. Cavaleiro, J. Elguero, M.L. Jimeno and A.M.S. Silva, *Chem Lett*.1991, 445.

13. A.G. Doshi, P.A. Soni and B.J. Ghiya. *Indian J. Chem.* 1986, **25,** B759.
14. J.K. Makrandi and Seema. *Indian J. Chem.*1991, **72**, 201.
15. J. Iqbal, W. Fatima, W.A. Shaida, W. Rahma. *J.Chem. Res.*1982*,* **(s) 92**.
16. S.C. Kushawaha, Dinkar and J.B. Lal. *Indian J.Chem*. 1967, **5**(2), 82.
17. Z.B. Arian and H.J. Suschitzkyl. *J. Chem*. 1961, 2242.
18. R. Kuhn and H.R. Hensel. *Chem. Bar*. 1953, **86**, 1333.
19. P.F. Devitt, A. Timoney and M.A. Vickars. *J. Org. Chem.* 1961, **26**, 4941.
20. N.P. Buu- Hoi, N.D. Xuong. *Bull. Soc. Chim. France*, 1956, 1646.
21. Y.S. Agashimuddin, S.D. Jolad. *Indian J. Chem.*1965, **3**(S), 220.

*** Corresponding author: S.B. Pawar;** P.G. Department of Chemistry, Shri Anand College Pathardi, Ahmednagar. Maharashtra, India