*International Journal of Theoretical and Applied Science* **3**(2): 13-14(2011) **ISSN No. (Print) : 0975-1718**
**ISSN No. (Online) : 2249-3247**

# Spectroscopic (FTIR, 1h-NMR and Mass) Studies of New Dihydropyrimidine Derivatives

***K. Thakur* and J. Trivedi*****

**Department of Chemistry, Shoolini University of Biotechnology and Management Sciences, Solan, (Himachal Pradesh)*
***Department of Pharmaceutical Chemistry, ISF College of Pharmacy, Ferozpur Road, (PB) India*

*(Received 20 October, 2011, Accepted 24 November, 2011)*

**ABSTRACT : Dihydropyrimidines (DHPMs) are of much importance due to excellent pharmacological properties. In this account we have synthesized new functionalized DHPMs (a) 5-benzoyl-6-methyl-4-phenyl-3,4-dihydropyrimidin-2(1H)-one (KT-01) and (b) 5-benzoyl-4-(4-chlorophenyl)-6-methyl-3,4-dihydropyrimidin-2(1H)-one (KT-03) in two step reaction. The first step involves the synthesis of benzoylacetone and the second step involves the one pot, three component cyclo condensation reaction of benzoylacetone, benzaldehyde (substituted) and urea using catalytic amount of concentrated HCl in ethanol provided access to targeted dihydropyrimidine. Structures of these compounds have been confirmed by IR, $^1$H-NMR and Mass spectroscopy.**

**Keywords:** Multicomponent reactions; Dihydropyrimidines ; FTIR, 1H-NMR and Mass spectroscopy.

## 1. INTRODUCTION

Multicomponent reactions (MCRs) are of increasing importance in the organic and medicinal chemistry for various reasons [1] such as their high degree of atom economy, applications in the combinatorial chemistry and diversity-oriented synthesis [2]. In MCRs three or more reactants come together in a single reaction vessel to form a new product that contain portion of all the components. One prominent MCR that produces an interesting class of nitrogen heterocycles is the Biginelli dihydropyrimidine synthesis.

Nitrogen heterocycles are abundant in nature and are of great significance to life because their structural subunits exist in many natural products such as vitamins, hormones, antibiotics, and alkaloids, as well as pharmaceuticals, herbicides, dyes, and many more compounds [3].

In 1983, Italian chemist Pietro Biginelli [4] reported the acid catalyzed cyclocondensation reaction of a β-ketoester (1), aldehyde (2) and urea (3) or thiourea , a procedure known as the Biginelli reaction is receiving increasing attention. The reaction was carried out by simple heating a mixture of three components dissolved in ethanol with a catalytic amount of HCl at reflux temperature. The product of this novel one-pot, three-component synthesis that precipitated on cooling of the reaction mixture was identified correctly by Biginelli as 3,4-dihydropyrimidine-2(1*H*)-one **4** (Fig. 1) [5].

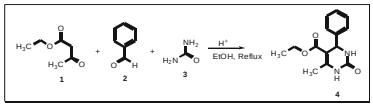



Fig. 1: Biginelli reaction

In the present work, we wish to report new dihydropyrimidines using Beginelli reaction. Structures of these compounds have been confirmed by FTIR, 1H-NMR and Mass spectroscopy.

## II. MATERIALS AND METHODS

Melting points were determined in a melting point apparatus (Sentwin India). The TLC of the compounds were performed on silica gel G coated glass plate with solvent system as ethylacetate: hexane (6 : 4). Chemicals used in this study are of analytical grade and some of them are used with further purification. IR spectra of a synthesized compound was obtained by preparing KBr pellet, using Perkin Elmer Spectrum 400 FT-IR spectrophotometer. $^1$H-NMR studies were done on Avance-11 Bruker FT-NMR spectrophotometer 300 MHz using DMSO. Mass spectra was recorded on Water *Q T* of Micro LS-MS Mass spectrophotometer.

### A. *Procedure for the Synthesis of Benzoylacetone:*

Benzoyl acetone was synthesized as per the method reported in Vogel textbook for Practical Organic Chemistry.

### B. *General Procedure for the Synthesis of DHPMs:*

Benzoylacetone 0.1 M, benzaldehyde 0.1M and urea 0.15 M were taken in a round bottom flask taking methanol as solvent and the contents were dissolved by gently heating the flask. conc. HCl (5-6 drops) was added to the flask and was then refluxed for 6-8 hrs and the completion of the reaction was monitored using TLC. After cooling at the room temperature the solid crystalline product was filtered and washed with chilled methanol and then recrystallized from ethanol to afford the new DHPMs (Fig. 2).

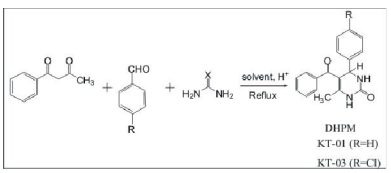



Fig. 2: Synthesis of DHPMs

## III. RESULTS AND DISCUSSION

The dihydropyrimidines were synthesized as per the reported Beginelli method. The physical data are given in Table 1. The structure of KT-01 and KT-03 are confirmed by FT-IR, 1H-NMR and Mass spectroscopy.

**Table 1. Physical properties of DHPMs**

| Code | MolFormula | Mol.Wt. | M. P. | $R_f$ Value | % Yield |
|---|---|---|---|---|---|
| KT-01 | $C_{18}H_{16}N_2O_2$ | 292.3 | 210-213°C | 0.5 | 67 |
| KT-03 | $C_{18}H_{15}ClN_2O_2$ | 326.7 | 236-238°C | 0.53 | 70 |

**5-benzoyl-6-methyl-4-phenyl-3,4 dihydropyrimidin-2(1H)-one (KT-01)**

**IR (KBr) $cm^{-1}$ (Fig. 3):** 3297 (N-H str.), 3108 (C-H str.), 1691 (>C = O), 1576, 1457,1427 (ring skeleton), 1330 ($CH_3$).

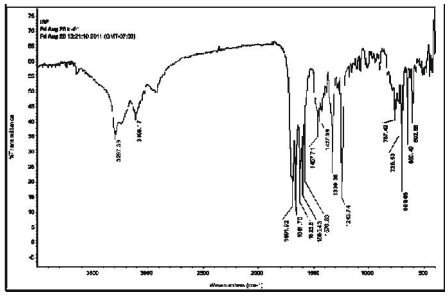
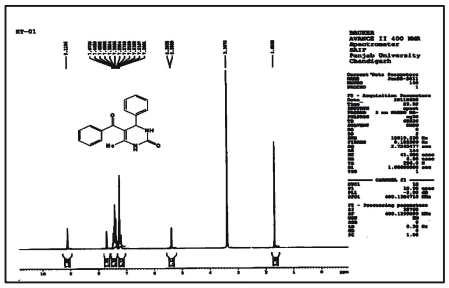


Fig. 3: FTIR Spectrum of KT-01 Fig.

**$^1$H NMR 300 MHz (DMSO-$d_6$, ä ppm) (Fig. 4):** 1.69 (s, 3H, $CH_3$), 5.39 (s, 1H, CH), 7.20-7.47 (m, 10H, Ar-H), 7.71 (s, 1H, NH), 9.11 (s, 1H, NH).



4: $^1$H- NMR Spectrum of KT-01

**Mass: [m/e (%)], M. Wt. (Fig. 5):** 293.1 ($M^+$), 250.1, 167.1

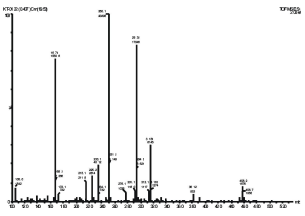
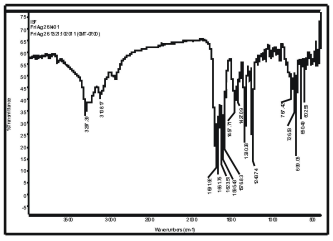


Fig. 5: Mass Spectrum of KT-01 Fig.

5-benzoyl-4-(4-chlorophenyl)-6-methyl-3,4 dihydropyrimidin-2(1H)-one (KT-03)

**IR (KBr) $cm^{-1}$ (Fig. 6):** 3278 (N-H str.), 2927 (C-H str.), 1718 (>C=O), 1567, 1444, 1420 (ring skeleton), 1355 ($CH_3$).



6: FTIR Spectrum of KT-03

**$^1$H NMR 300 MHz (DMSO-$d_6$, ä ppm) (Fig. 7):** 1.70 (s, 3H, $CH_3$), 5.41 (s, 1H, CH), 7.2-7.6 (m, 5H, Ar-H), 7.25 (d, 4H, Ar-H), 7.70 (s, 1H, NH), 9.1 (s, 1H, NH).

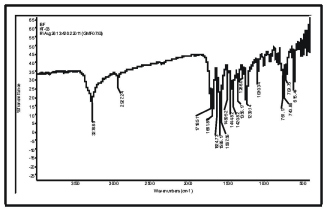
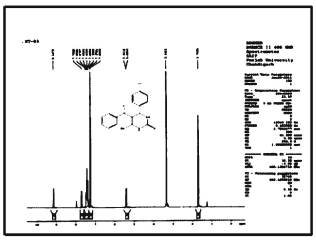


Fig. 7: $^1$H-NMR Spectrum of KT-03 Fig.

**Mass: [m/e (%)], M. Wt. (Fig. 8):** 327.1($M^+$), 284.1, 201.1



8: Mass Spectrum of KT-03

## IV. ACKNOWLEDGEMENTS

The authors thank Prof. P. K. Khosla, H'ble Vice-Chancellor, Shoolini University, Solan, for providing adequate infrastructure and financial support for the analytical characterization of the compound. Authers are also thankful to Punjab University, Chandigarh, for providing FT-IR, $^1$H NMR and Mass also ISF College of Pharmacy, Ferozpur for FT-IR Spectroscopy facilities.

## REFERENCES

[1] L. F. Tietze and M. E. Lieb, *Curr. Opin. Chem. Biol.* **2**: 363(1998).

[2] D. J. Ramo and M. Yus, *Angew. Chem.* **44**: 1602 (2005).

[3] Padwa, Bur. *Chem. Rev.* **104**: 2401(2004).

[4] P. Biginelli, *Gazz. Chim. Ital.* **23**: 366(1893).

[5] O. Kappe, *Tetrahedron.* **49**: 6937(1993).

[6] B. S. Furniss, A. J. Hannaford, In *Vogel's text book of practical organic chemistry* (Addison-Wesley Longman, Harlow, 1998).