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Preparation and Spectroscopic Study of the Reaction of 4-Nitroacetophenone , Furfural and Thiourea

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Abstract
The Chalcone (1) were prepared by Claisen –Schmidt condensation of 4-nitroacetophenone with furfural in presence of sodium hydroxide and ethanol. This chalcone were treated with thiourea, guanidine hydrochloride to yield the respective pyrimidine derivative.
The synthesized compounds were characterized by UV, IR, 1H-NMR & 13C-NMR spectral data.
Keywords: Chalcones, Furfural, 4-nitroacetophenone,.

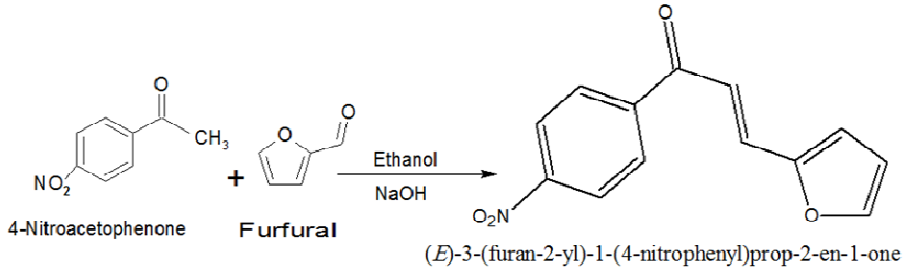
1. Introduction

Heterocyclic compounds are important to human life because their structural subunits exist in many natural products such as vitamins, hormones, antibiotics and pigments [1,2]. Pyrazoles are five member ring heterocyclic compounds having some structural features with two nitrogen atoms in adjacent position [3]. The best described property of almost are pyrazoles is in the treatment of inflammation and inflammation associated disorder, such as arthritis [4]. Pyrazole derivatives are the subject of many research studies due to their widespread potential biological activities such as antimicrobial [5, 6], antiviral [7], antioxidant [8], antitumor [9,10] , antihistaminic [11], antidepressant [12] and fungicides [13]. Several pyrazole derivatives have been found to possess significant activities such as ACE inhibitor [14], anttiproliferative [15], anti-inflammatory [16] and antiprotozoal [17, 18] which render them valuable active ingredients of medicine and plant protecting agents. Further current literature indicates 1,2 –pyrazole derivatives to possess diverse biological activities [19]. These compounds are useful in the field of medicine and are used as a starting material for the synthesis of new drugs [20-29]. In view of these data we have undertaken the synthesis, characterization and antimicrobial evaluation of substituted pyrazoles. All the synthesized compounds were characterized on the basis of IR, 1H & 13C NMR spectral data and elemental analysis. The physical data of titled compounds are summarized and presented in the result and discussion part.

2. Experimental section

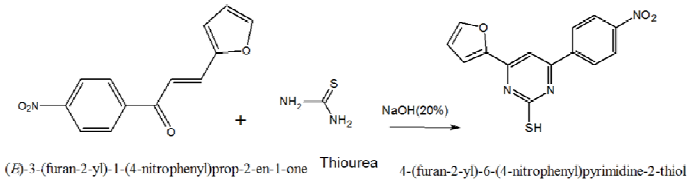
2.1. Materials and Methods:

The melting points were carried out in the open capillary tube and were uncorrected. Thin layer chromatography was performed using silica gel coated on a glass plate and pots were visualized by exposure to iodine vapour. IR spectra of compounds were scanned on Shimadzu IR spectrophotometer using KBr disc and expressed in cm-1.
1Hand 13C NMR spectra were recorded in DMSO-D6 on BRUKER (400MHz) spectrometer using TMS as an internal standard (chemical shifts in δ, ppm. The synthesis of the targeted compound was accomplished according to the reaction sequence illustrated in Scheme1 and Scheme 2.
2- Synthesis of 3-(furan-2-yl)-1-(4-nitrophenyl)prop-2-en-1-one (FNBA):
A mixture of 4-nitroacetophenone (0.01 mol) and furfural (0.01 mol) is dissolved in ethanoic NaOH (20ml) was stirred for about 3 h with a mechanical stirrer and kept in a refrigerator for 24 h. The content is poured into crushed ice and acidified with HCl. The product formed was filtered washed with water and recrystallized from ethanol to give compound FNBA (yield 85%, melting point = 203-205 °C).



Scheme 1: Reaction of 4-nitroacetophenone with furfura.

3 - Synthesis of pyrmidine derivative (TPFN):
A mixture of 3-(furan-2-yl)-1-(4-nitrophenyl)prop-2-en-1-one (0.02mol), thiourea (0.02 mol) were dissolved in ethanolic sodium hydroxide (10ml) was reflux overnight. The precipitate obtained was filtered, washed and recrystallized from ethanol to give compound TPFN (yield 74%, melting point = 180-182°C).



Scheme 2: Reaction of FNBA with thiourea

Table1 show Some properties of the synthesized of chalcone and pyrmidine derivative.

Table 1:

Compounds	Formulas	Color	Mol.W gr/mol	m.p °C	Yield (%)
chalcone	$C_{13}H_9O_4N$	yellow	243	203- 205	85%
pyrmidine	$C_{14}H9N_3O_2S$	dark brown	283	180-182	74%

3.Result and discussion:

IR spectra of 3-(furan-2-yl)-1-(4-nitrophenyl)prop-2-en-1-one (chalcone):

The infrared spectra for the present compounds taken in the range 400-4000 cm^{-1} help to indicate regions of absorption vibrations. . The main stretching modes are for ν(C=O), ν(C=C) and ν(NO_2).

The IR data of the spectra of 3-(furan-2-yl)-1-(4-nitrophenyl)prop-2-en-1-one (chalcone) and4-(furan-2-yl)-6-(4-nitrophenyl)pyrimidine-2-thiol(pyrmidine derivative) are presented in Table 2,3.

Spectrum of Chalcone shows a sharp band at (1720cm^{-1}) due to ν(C=O),3100 (aromatic C-H), 2885 (aliphatic C-H), 1601(aromaticC=C), 1450 (aliphatic C=C), 1350 (NO_2).

The IR data of the spectra of 4-(furan-2-yl)-6-(4-nitrophenyl)pyrimidine-2-thiol:

a sharp band shows at (1602cm^{-1}) due to ν(C=N) , 1272 cm-1 ν (C-N), 3100 (aromatic C-H), 2885 (aliphatic C-H), 1573(aromaticC=C),2400cm^{-1} ν(SH),1450 (aliphatic C=C), 1350 (NO_2).

Table 2. Wave number (cm-1) of the functional groups of Chalcone:

Functional Group of chalcone	Wave number [cm^{-1}]
C=O	1720
aromatic C-H	3100
aliphatic C-H	2885
aromaticC=C	1601
aliphatic C=C	1450
NO_2	1350

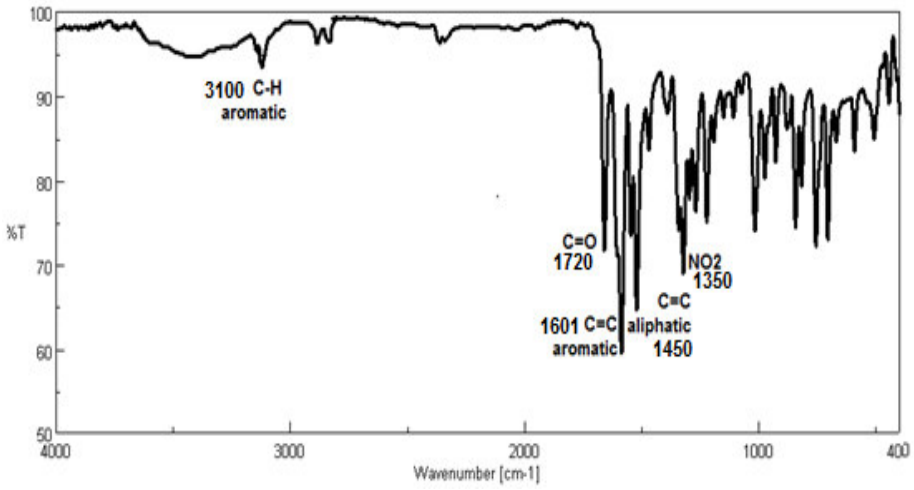


Figure 1: IR spectrum of chalcone.

Table 2. Wave number (cm-1) of the functional groups of (TPFN):

Functional Group of pyrmidine	Wave number [cm^{-1}]
C=N	1602
C-N	1272
SH	2400
aromatic C-H	3100
aliphatic C-H	2820
aromaticC=C	1570
NO_2	1350

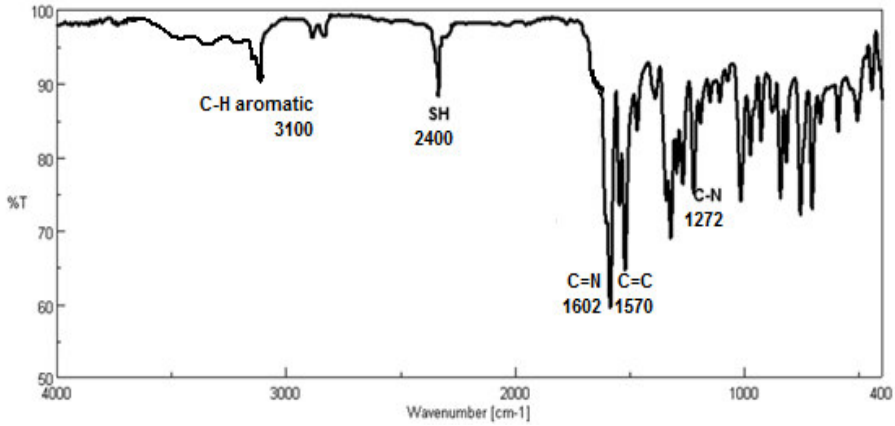


Figure 2: IR spectrum of pyrmidine.

The ^{1}H-NMR spectrum of chalchone (FNBA) (Figure 3) and of chemical shifts showed in Table 3

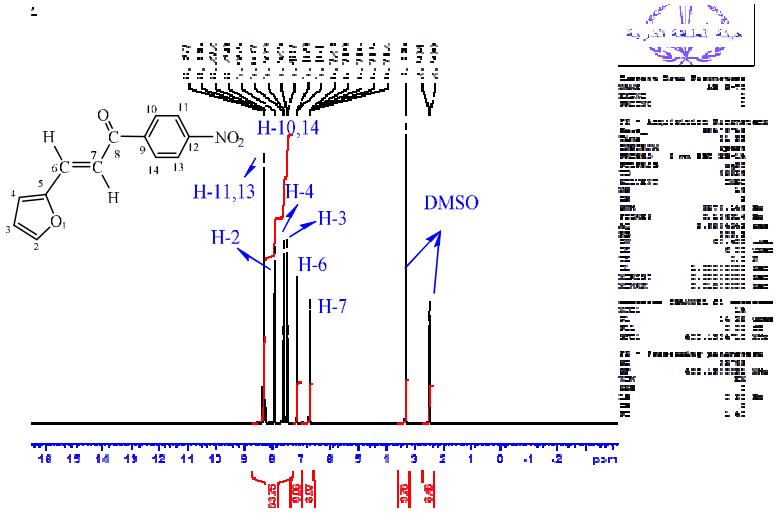


Figure 3: 1H-NMR spectrum of chalchone (FNBA).

Table 3. The (1H-NMR) chemical shifts (ppm) of chalcone (FNBA).

J [Hz]	chemical shift [PPM]	proton number	
-	3.336	(S)	1
8	7.955	(H,d)	2
8	7.525	(H,t)	3
8	7.699	(H,d)	4
8	7.169	(H,d)	6
8	6.723	(H,d)	7
8	8.20	(H,d)	10
8	8.336	(H,d)	11
8	8.357	(H,d)	13
8	8.212	(H,d)	14

The ^{13}C NMR spectrum of chalcone (FNBA) (Figure 4) showed 11 signals; that shows (δ=188 ppm) for carbonyl group(C=O) , and of the chemical other shifts showed in Table 4

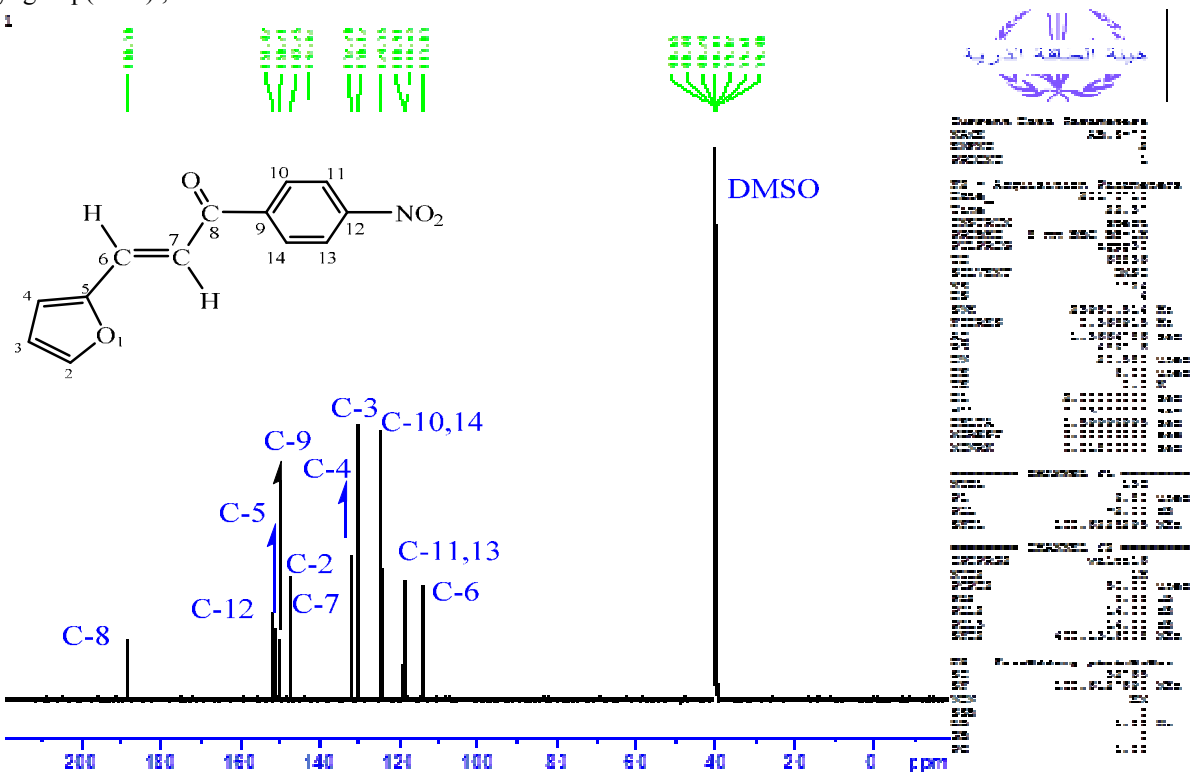


Figure 4: ^{13}C-NMR spectrum of spectrum of chalchone (FNBA).

Table 4. The (^{13}C -NMR) chemical shifts (ppm) of chalcone (FNBA).

chemical shift [PPM]	.Carbon number
149	2
130	3
138	4
152	5
115	6
145	7
188	8
144	9
125	10
120	11
153	12
119	13
125	14

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4. Reference

[1] Yadav, P. S; Devpraksh; Senthilkumar, G. P. Inter. J. Pharm. Sci. Drug Res., 2011, 3(1), 1.
[2] Yuhong, J. U; Varma, R. S. J. Org. Chem., 2006, 71, 135.
[3] Jamwel, A; Javed,A; Bhardwaj,V; J. Pharm. BioSci., 2013, 3, 114.
[4] John J, Tally Donald J and Rogier Jr, Both of St, Louis, Mo G D. Searle &.Co,. Skokie, 1995, Pt No: 5, 434, 178

[5] Pimerova, E.V; Voronina, E.V; Pharm. Chem. J., 2001, 35, 18.
[6] Kavitha, R; Nagoor Meeran, M; Sureshjeyakumar, R.P; Chem Sci Trans., 2015, 4(4), 1001.
[7] Janus, S.L;Magdif, A.Z; Erik, B.P; Claus, N; Chem., 1999, 130, 1167.
[7] Pasin,J.S.M; Ferreia,A.P.O; Saraiva, A.L.L; Ratzlaff, V; Andrighetto, R; Machado, P; Marchesam, S; Zanette, R.A; Martins, M.A.P; Braz J. Med BioI Res., 2010, 43, 1193.
[9] Park, H.J; Lee, K; Park, S; Ahn, B; Lee, J.C; Cho, H.Y; Lee, K.I; Bioorg. Med. Chem. Lett., 2005, 15, 3307.
[10] Bouabdallah, I; M'barek, L.A; Zyad, A; Ramadan, A; Zidane, I; Melhaoui, A; Nat. Prod. Res., 2006, 20, 1024.
[11] Yildirim, I; Ozdemir, N; Akçamur, Y; Dinçer, M; Andaç, O; Acta Cryst., 2005, E61, 256.
[12] Bailey, D.M; Hansen, P.E; Hlavac, A.G; Baizman, E.R; Pearl, J; Defelice, A.F; Feigenson, M.E; J. Med.Chem., 1985, 28, 256.
[13] Chu, C.K; Cutler, J; J. Heterocycl. Chem., 1986, 23, 289.
[14] Bonsei, M; Loizzo, M.R; Statti, G. A; Michel, S; Tilequin, F; Mencichini, F; Bioorg. Med. Chem. Lett., 2010,20, 1990.
[15] Chimichi, S; Boccalini, M; Hassan, M.M.M; Viola, G; Acqua, F.D; Curini, M; Tetrahedron, 2006, 62, 90.
[16] Nugent Richard, Marphy Meghan J. Med. Chem., 1993, 36 (1), 134.
[17] Hantoon, M.A; Minnesota Medicine, 2001, 84, 102.
[18] Zhang, X; Li, X; Allan, G. F; Sbriscia, T; J Med Chem., 2007, 50(16), 3857.
[19] Abunada, N. M; Hasaneen, H. M; Kandile, N.G; Miqdad, O. A; Molecules, 2008, 13(7), 1501.
[20] Smith, I. K; Time, 2000, 155(16), 89.
[21] Mao, Y; Mao, X; Faming Zhu Shenq Gong Shuom., 2003, 23, 468.
[22] Tang, X; Serizawa, A; Tokunaga, M; Yasuda, M; Matsushita, K; Terachi, T; Osamura, R;Human Patho., 2006,37, 1187.
[23] Mohsin, M; J Med Sci., 2008, 1(1), 42.
[24] Kato M; Ayumi., 2009, 230, 458.
[25] Priddy, D. B; Franks, M; Konas, M; Vrana, M. A; Yoon, T, H; McGrath, J, E; Polym Preprints., 1993, 34, 310.
[26] Khananashvili, L; Markarashvili, E; Vardosanidze, V; Tkeshelashvili, R; Butskhrikidze, B; Nogaideli, G; Tsomaia, N; Izv Akad Nauk Gruzii., 2001, 27, 48.
[27] Guan, W; Li, X; Xiao, S; Zhong Yao Yu Linch., 2005, 5, 283.
[28] Ackermann, L; Althammer, A; Mayer, P; Synthesis, 2009, 20, 3493.
[29] Sammour, A. E. A; Tetrahedron, 1967, 20(4), 1067.