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SYNTHESIS OF NOVEL FURAN AND THEIR HYDRAZONES

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Abstract
β-Benzoylproponic acid **(1)** on lactonization reaction with excess of acetic anhydride, and few drops of concentrated sulfuric acid as a catalyst reviled 5-phenyl-furan-2(3H)-ones **(2)**. A mixture of 5-phenyl-furan-2(3H)-ones with aromatic aldehydes, potassium hydroxide as a catalyst result to 2-Phenyl-4-benzylidene-5-hydrazinofuran-2-ene (**3**), which on further reaction with hydrazine hydrates yielded corresponding 5-hydrazine (**4)**. This on condensation reaction with different aldehydes reviled final 2-Phenyl-4-benzylidene-5-benzylidinehydrazo-furan-2-ene (**5)**. The structures of the compounds have been elucidated on the basis of spectral analysis.

Keywords: β-benzoylproponic acid, furan, aromatic aldehydes.

Introduction

Substituted furan derivatives are found to be associated with various biological activities such as anti-bacterial[1], anti-tumorigenic acticity[2], anti-muscarinic agent[3], anti-platelet[4], anti-phage activity[5], and important intermediates for synthesis of molecules having wide therapeutic application[6]. Therefore, there is considerable interest of having available efficient routes to these heterocycles and better understand their reactivity.

The ability of α, β-unsaturated ketones to react with various nucelophilic reagents prompted us to synthesis some new fused compounds.

Experimental Section

Melting points of all synthesized compounds were determined in open capillary tubes on an electrothermal apparatus and are uncorrected. The purity of the compounds was monitored by thin layer chromatography on silica gel coated aluminium plates (Merck) as adsorbent and UV light as visualizing agent. IR spectra (KBr in cm-1) were recorded on a Perkin-Elmer spectrophotometer in the range of 4000-400 cm-1. 1H NMR spectra were recorded on a Varian 500 MHz NMR spectrometer using $CDCl_3$/DMSO-d6 as solvent and TMS as an internal standard (chemical shifts in δppm).

5-Phenyl-3-benzylidene-furan-4-ene-2-one (3)

General procedure

Lactonization of β-benzoylproponic acid (17.8gm, 0.1 moles) with excess of acetic anhydride (10ml) and few drop of Conc. Sulfuric acid as a catalyst, stirring to clear solution and then mixture was added with vigorous stirring to 250ml of cold water chilled in an ice bath stirred for 30 minutes, filter and solid washed with 75ml water recrystallized form 95% ethanol yielded 71% furan ring **(2**). This on stirring with different aldehydes in ethanol in presence of triethylamine a catalyst reviled solid product, filter and washed with ice cold ethanol to yield **(3)**.

2-Phenyl-4-benzylidene-5-hydrazino furan-2-ene (4)

General procedure

Mixture of **2** (0.05 mole), Hydrazine hydrate (0.05 mole), in Methanol 10 mL as a solvent was taken in 100 mL round bottom flask and the mixture was reflux for 1 hrs. After monitoring the reaction on TLC, the reaction mixture cooled and dumped on to the ice, filtered and recrystallized from ethanol to yield **4.**

2-Phenyl-4-benzylidene-5arylhydrazo-furan-2-ene (5)

General procedure

Mixture of **3** (0.05 mole), aldehydes (0.05 mole), Potassium hydroxide (0.1 mole), in Ethanol 10 mL as a solvent was taken in 100 mL round bottom flask and the mixture was reflux for 4 hrs. After monitoring the reaction on TLC, the reaction mixture cooled and dumped on to the ice, filtered and recrystallized from ethanol to yield **5.**

Representative spectral data

2-Phenyl-4-benzylidine-5-(3-hydroxy)benzylidihydro-furan-2-ene (5a)

m.p; 178-80°C; IR (Vmax): 3370, 3084, 1612cm^{-1}; ^{1}HNMR (500 MHz, DMSO-d_6) δ= 6.80-7.88(m, 16H, ArH & 2CH), 8.90(bs,1H, OH); ^{13}CNMR (500 MHz, DMSO-d_6) 112.84(CH), 120.12(C=C), 124.52-133.92(Ar-C), 148.05(C=N), 150.12(C=N); GC.MS: m/z= 366(M^+); Anal. Calcd. for $C_{24}H_{18}N_2O_2$: C, 78.69. H, 4.92. N, 7.65. Found: C, 78.67. H, 4.88. N, 7.62.

2-Phenyl-4(4'-methoxy)-benzylidine-5-benzylidihydrazo-furan-2-ene (5b)

M.P; 170-72°C; IR (Vmax): 3062, 1628cm^{-1}; ^{1}HNMR (500 MHz, DMSO-d_6) δ= 3.50(s, 3H, OCH_3), 6.30-7.87(m, 16H, ArH & 2CH). Anal. Calcd. for $C_{25}H_{20}N_2O_2$: C, 78.95. H, 5.26. N, 7.37. Found: C, 78.91. H, 5.23. N, 7.34.

2-Phenyl-4(3'-methoxy-4'-hydroxy)-benzylidine-5(3''-methoxy-4''-hydroxy)-benzylidihydrazo-furan-2-ene (5c)

M.P; 168-70°C; IR (Vmax): 3475, 3072, 1628cm^{-1}; ^{1}HNMR (500 MHz, DMSO-d_6) δ= 3.70(s, 3H, OCH_3), 3.80(s, 3H, OCH_3), 6.60-7.75(m, 13H, ArH & 2CH), 8.80(bs, 1H, OH), 9.80(bs, 1H, OH). Anal.Calcd. for $C_{26}H_{22}N_2O_5$: C, 70.59. H, 4.98. N, 6.33. Found: C, 70.56. H, 4.92. N, 6.30.

Antimicrobial and antifungal activity

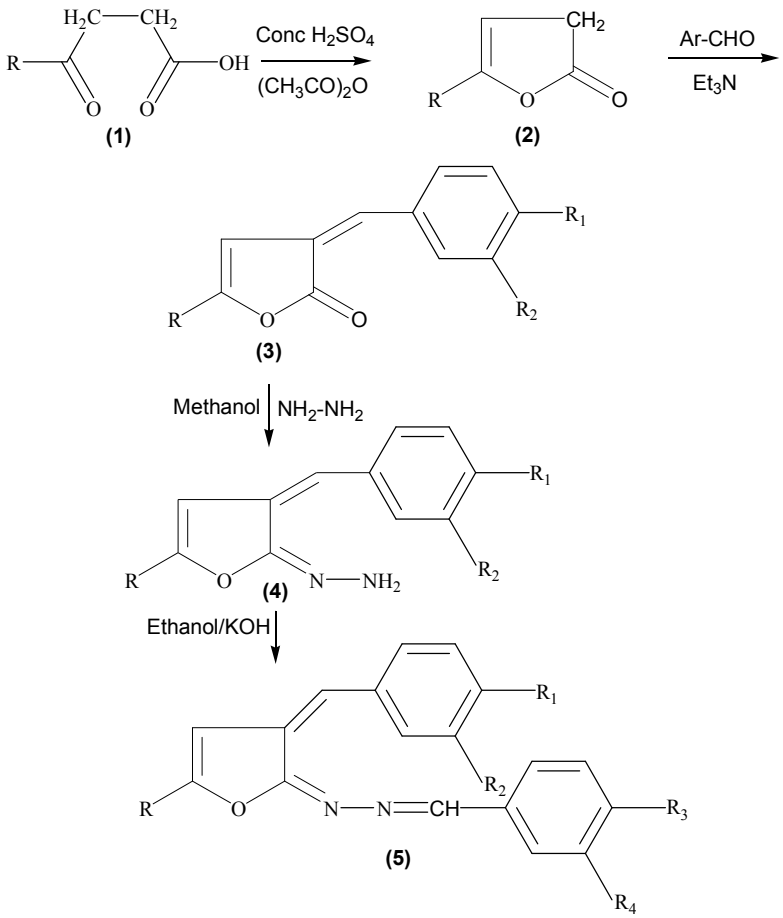
Representative samples were screened for their antimicrobial and antifungal activity against gram-negative bacteria, E coli and P aeruginosa and gram-positive bacteria, S aureus, and C

diphtheriae using disc diffusion method[7, 8]. The zone of inhibition was measured in mm and the activity was compared with standard drug. The results of antibacterial screening studies are reported in **Table I**.

Table I — Antibacterial activity of compound **5**				
Compounds	Zone of Inhibition (in mm)			
	Gram Positive		Gram Negative	
	S.aureus	*C.diphtheria*	*P.aeruginosa*	E.coli
5a	18	20	10	11
5c	22	20	10	12
Ampicillin trihydrate	26	28	24	21
DMSO	00	00	00	00

* Diameter of the disc was 6mm, concentration of the compounds taken was about 100 µg/mL.

Scheme-I



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Reference

1 L. A. Safronova, G. K. Kornirnko, G. M. Shub and A. D. Shebaldova, Pharam Chem Journ, **34** (2000), 476.
2 M. Trushule, E. Kupche, I. Augustane, N. V. Verovskii, E. Lukevits, L. Baumane, R. Gavar and Ya. Stradyn, Chememistry of Heterocyclic compound, **17**,(1991), 1358-64.
3 James F. Stubbins, Patrica M. Hudgins and Dagmar C. Murphy, Jouranl of Pharmacetical science .**69** (1980), 534.
4 Kyung-Sup Lee, Jung-Woo Park, Yong-Ri Jin, In-Sang Jung, Mi-Ra Cho, Kyu-Yang Yi, Sung-Eun Yoo, Hun-Jong Chung, Yeo-Pyo Yun, Tae-Kyu Park and Hwa-Sup Shin. Arch Pharm Res, **29** (2006), 375-383.
5 T. G. Nikolaeva, A. A Safonova and A. P.Kriven Ko, Pharmacetical Chemistry Jouranl. **31** (1997) 476.
6 Jitendra M. Gajera, Balasubramanian Gopalan, Pravin S. Yadav, Sandip D Patil, Laxmikant A. Gharat, J. Heterocycilc Chem, **45** (2008) 797.
7 Cruickshank R, Duguid J P & Marmion B P, Medicinal Microbiology,12th edn, Vol 11, 1975, (Churchill Livingstone, London).
8 Arthington-Skaggs B A, Motley M & Morrison C J, J Clin Microbiology, 38, 2000, 2254.

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