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Synthesis of iodo-nitro-chalcones

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ABSTRACT

Six different Chalcones I(a)-I(f) were synthesized by condensing 2-hydroxy-3-iodo-5-methyl acetophenone with six different aromatic aldehydes in ethanol using NaOH. The synthesized compounds were characterized by I.R., NMR spectral analysis.

Key Words: - iodo – nitro-Chalcones.

INTRODUCTION

Claisen synthesized Chalcones by condensation of 2-hydroxyacetophenone with aromatic aldehydes in presence of acidic [1] or basic [2] media. Said Eddrir et al have studided an efficient synthesis of Chalcones based on the Suzuki reaction [3]. Ognyan Petrov et al have studied SOCl2/ EtOH: catalytic system for synthesis of chalcones [4]. Fang Dong et al have studied a synthesis of Chalcones via claisen- Schmidt condensation of reaction catalyzed by acyclic acidic ionic liquids [5].M.Lkshamani Kantam et al have synthesized the effective sunthesis of Chalcones by a solid base catalyst [6].Mao Sheng Cheng et al have synthesized a solid phase synthesis of Chalcones by claisen- Schmidt condensation [7].

Qiong Xu and etal synthesized the chalcones catalysed by a novel solid sulfonic acid from Bamboo [8]. G.Venkat Reddy et al have studied microwave assisted knowengel condensation : A facile method for the synthesis of Chalcones [9]. Karsten Krohn have studied the isolation and synthesis of Chalcones with different degree of saturation [10]. D.M.Pore have synthesized the efficient synthesis of Chalcones at room temperature in presence of potassium phosphate [11]. Nora M.Rateb have synthesized the atom-efficient, solvent-free, green synthesis of Chalcones by grinding [12]. Bhagyesh Baviskar et al have studied design and synthesis of of some new novel Chalcones as potent antimicrobial agent [13]. Shoji Shibata have studied anti-tumorignic Chalcones [14]. Yi Xia et al have studied antitumor agents part:202 novel 2'- amino Chalcones : designs, synthesis and biological evaluation.[15]. Said Sebti et al have studied dramatic activity enhancement of natural phosphate catalyst by lithium nitrate. An efficient synthesis of Chalcones [16]. Shailesh H. Shah have studied synthesis , charecterisation and antimicrobial activity of some novel chalcones [17].

Chandrashekhar C. H. have studied synthesis and antimicrobial activity of chalcones of naphtho [2,1-b] condensed with barbituric acid [18]. Ramesh C. Kumboj studied eco-friendly synthesis and antimicrobial activity of chalcones [19]. S.S. Mokale have studied the synthesis of some new biologically active chalcones and flavanones [20]. Hemendra Pratap Singh have studied synthesis and pharmacological screening of some novel chloro chalconesemicarbazone derivatives [21].

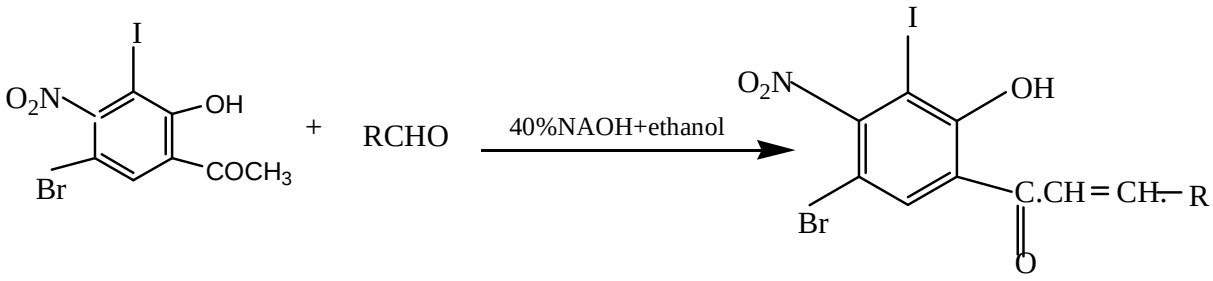
MATERIALS AND METHODS

Melting points of all synthesized compounds were determined in open capillary tube and are uncorrected. The purity of compounds was checked by TLC using silica G. IR. Spectra were recorded on Perkin-Climer-841 spectrphotoometer (Cm^{-1}) in KBr disc and NMR (Brucker Avance II 400 NMR) using $CDCl_3$ as solvent.

Synthesis of 2-hydroxy-3-iodo-5-methyl-acetophenone (Compound-I)
By known method from p-bromo phenol to p-bromo-acetate prepared and then by fries migration-2-hydroxy-5-bromo acetophenone which on iodination gives 2-hydroxy-3-iodo-5-bromo acetophenone (Comp-1) which on nitration gives 2-hydroxy-3-iodo-4-nitro acetophenone (comp-**I**)

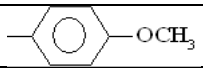
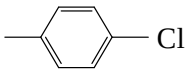
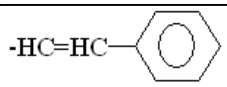
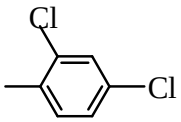
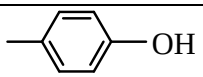
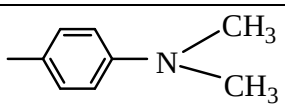
Synthesis of substituted 2-hydroxy-3-iodo-4-nitro-5-bromo Chalcones [$I_{(a)} - I_{(f)}$]
Compound $I_{(a)}$to $I_{(f)}$were synthesized from2-hydroxy-3-iodo-4-nitro-5-bromo acetophenone by reacting with six different aromatic aldehydes by known method in solvent ethanol using 40% NaOH. The physical data of compounds $I_{(a)}$ to $I_{(f)}$ is given in table

Reaction Scheme :



(I) **($I_a - I_F$)**

The groups R are shown in Table.

S. N.	Compound No.	R	Mole. Formula	M.P.^{0}C	Yield
1.	I_a		$C_{16}H_{11}O_5I\ N\ Br$	146 ^{0}C	72%
2.	I_b		$C_{15}H_8O_4INBrCl$	158 ^{a}C	68%
3.	I_c		$C_{17}H_{11}O_4INBr$	134 ^{a}C	70%
4.	I_d		$C_{15}H_7O_4INBrCl_2$	110 ^{a}C	66%
5.	I_e		$C_{15}H_9O_5INBr$	120 ^{a}C	60%
6.	I_f		$C_{17}H_{14}O_4IN_2Br$	188 ^{a}C	70%

RESULTS AND DISCUSSION

Compound $I_{(a)}$ – $I_{(f)}$ were synthesized through the route as shown in general reactions R as shown in table no. 1 Similarly, physical data as shown in table no. 1 The synthesized compounds I,(I_a) and (I_b) were confirmed on the basis of IR, NMR spectral analysis.

Characterization data of compound
2-hydroxy-3-iodo-4-nitro-5-bromo acetophenone (I)
IR (KBr) ν max cm $^{-1}$
3434.06 cm^{-1} (br) – phenolic OH , 3056 cm^{-1} (s) – Aromatic C-H stretching , 2917 cm^{-1} (s) Aliphatic stretching of Ar-O-CH3, 1739.87 cm^{-1} C=O stretching , 1582 & 1335 cm^{-1} (S) stretching due to NO_2, 640 cm^{-1} C-Br stretching, 540 cm^{-1} C-I stretching .

H1 NMR: [δ $CDCl_3$]
2.7-2.8 δ (S , 3H ,Ar-CO-CH_3) , 7.8 δ (d, 1H , Ar-H) , 8.1 δ (d, 1H , Ar-OH).

– 2- hydroxy -3-iodo-4-nitro-5-bromo-4'-methoxy Chalcone. (I_a)
IR (KBr) ν max cm $^{-1}$
3393.06 cm^{-1} (br) – phenolic OH , 3056.49 cm^{-1} (s) – Ar- C-H stretching , 2858 cm^{-1} Aliphatic stretching of Ar-O-CH_3, 1628 cm^{-1} (s) C=O stretching , 1541 cm^{-1} (S) CH=CH- Stretching , 1509 & 1323-1305 cm^{-1} (s) stretching due to NO_2 , 695cm^{-1} (S) C-Br stretching, 537 cm^{-1} C-I stretching .

H1 NMR: [δ $CDCl_3$]
3.8 δ (S , 3H ,Ar- OCH_3) , 7.00 δ (d, 2H , -CH=CH) , 7.1-8.1 δ (m, 5H , Ar-H) , 8.5 δ (br, 1H , Ar-OH).

2- Hydroxy -3-iodo-4-nitro-5-bromo-4'-chloro Chalcone. (I_a)
IR (KBr) ν max cm $^{-1}$
3435 cm^{-1} (br) – phenolic OH , 2925 cm^{-1} (s) – Ar- C-H stretching , 1641 cm^{-1} (S) C=O stretching, 1563 cm^{-1} (S) O=C-CH=CH stretching , 1262 cm^{-1} (S) Ar-O stretching , 1490 & 1327 cm^{-1} (S) stretching due to NO_2 , 1178 cm^{-1} (S) C-O stretching in phenol ,493 cm^{-1} (S) C-I stretching, 657 cm^{-1} (S) C-Br stretching , 818 cm^{-1} (S) C-Cl stretching.

H1 NMR: [δ $CDCl_3$]
6.8-7.1 δ (d, 2H , CH_A & CH_B of-CH=CH) , 7.4-7.8 δ (m, 5H , Ar-H) , 8.5 δ (d, 1H , Ar-OH).

CONCLUSION

Present study describes the synthesis of Chalcones. Compounds were characterized by I.R. & N.M.R. spectral analysis.

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