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SYNTHESIS AND CHARACTERIZATION OF CHLOROSUBSTITUTED DIKETONES

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KEYWORDS: Chalcone , diketone.

ABSTRACT
A diketone or dione is a molecule containing two ketone groups.An important class of intermediate for synthesis of various heterocyclic compound. In present investigation cholosubstituted diketone were prepaired by using general known method. The purity of synthesized compounds were checked by the thin layer chromatography and the compound were characterized by using IR spectra.

INTRODUCTION
β-Diketones compounds, whose simplest and the most widely known member is pentane-2,4-dione (informally referred to as acetylacetone), due to their structure (the presence of two carbonyl groups separated with one carbon atom) they have a variety of very interesting and specific properties. Their unique feature is keto-enol tautomerism, the presence of the ketone and the enol forms in equilibrium. Due to the presence of two carbonyl groups, β-Diketones are valuable intermediate for many chemical synthesis. Various drugs containing the heterocyclic moieties, such as carbazole, thaizole pyrazole, isoxazole, and imidazole etc. are the proven drugs against various ailments and are synthesized via a diketone.

The diketone research being stimulated by the versatility of these compounds as their biological activities and act as a antitumor[1], anti-oxidant[2], anti-viral[3] and immunomodulatory activities4. Anticancer[5], anti-inflammatory[6] agent.

MATERIALS AND METHODS
All the laboratory chemicals and solvents required for the synthesis were used are of highest purity and commercially available. Melting points of all synthesised compounds were determined by melting point apparatus. The purity of synthesised compounds was checked by thin layer chromatography .IR spectra were recorded on FTIR spectrophotometer using KBr pallets.

Synthesis of substituted β-Diketones
The synthesis involves following steps:

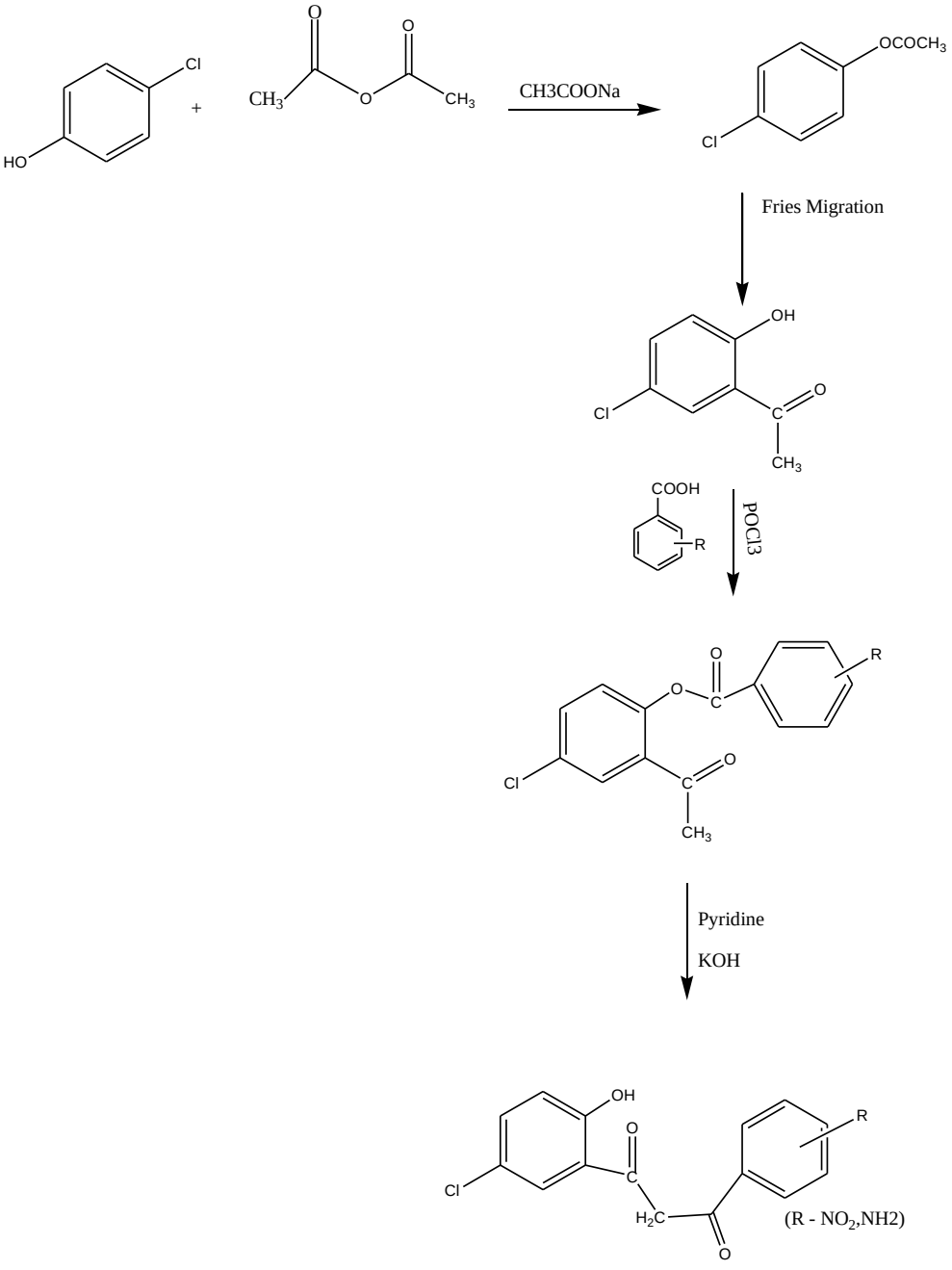
Preparation of 4-chloro-2-hydroxy-4 acetophenone-
p-chloro-phenol acetate was prepared by acetylation of p chloro-phenol.Then prepared p-chloro-phenyl acetate (50ml) and anhydrous aluminium chloride (120 g) were heated at 120^{0c} for 60 min. in an oil bath. The reaction mixture was cooled and quenched with acidified ice-cold water containing a little HCl to get Ketone.

Preparation of 2-benzoyloxy acetophenones
4-chloro-2-hydroxy acetophenone (1) (0.04 mol) and substituted benzoic acid (0.05 mol) were dissolved in pyridine and POCl3 is added drop by drop with constant stirring till the viscous mass is obtained. Maintain the temperature below 10^{0c} during the addition of POCl3 to the reaction mixture. The reaction mixture is allowed to stand for overnight at room temperature .The reaction mixture is decomposed by 10% HCl.The product thus separated was filtered, washed with water followed by sodium bicarbonate (10% solution) .The solid product was crystallised from ethanol .

Preparation of Substituted 1-(2'-hydroxy aryl)-3-aryl propane 1,3-diones (L1-L5)

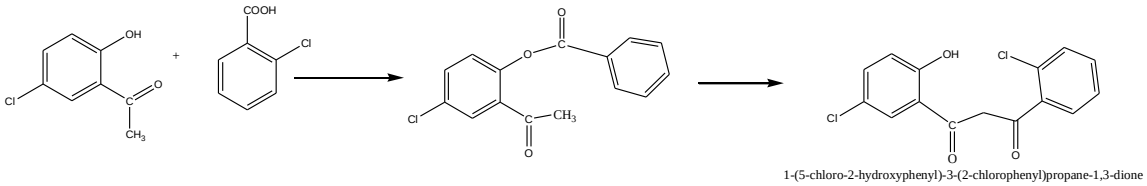
Substituted benzoyloxy acetophenones (0.05 mol) was dissolved in dry pyridine (40 ml). The solution was warmed up to about 60^{0c} and pulverised KOH (0.15 mol) was added slowly with constant stirring. After four hours the reaction mixture was acidified by adding ice cold dilute HCl (1:1). The solid product thus separated was filtered, washed with sodium bicarbonate solution (10%) and finally with water. It is then crystallised from ethanol acetic acid mixture to get 1-(2'-hydroxy aryl)-3-aryl propan-1-3- diones

General Reaction Scheme



Synthesis of -1-(5-chloro-2-hydroxyphenyl)-3-(2-chlorophenyl) propane-1,3-dione [L1]-

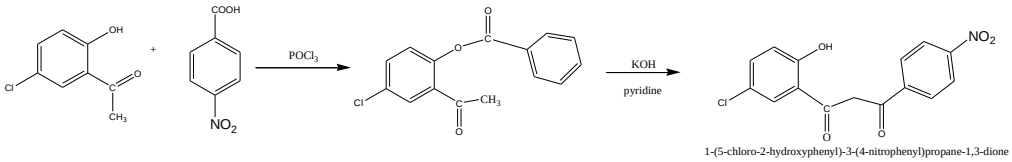
Chloro substituted benzoyloxy acetophenones prepaired from 4-chloro-2-hydroxy acetophenone (1) (0.04 mol) and chloro substituted benzoic acid (0.05 mol) was dissolved in dry pyridine (40 ml). The solution was warmed up to about 60^{0c} and pulverised KOH (0.15 mol) was added slowly with constant stirring. After four hours the reaction mixture was acidified by adding ice cold dilute HCl (1:1). The solid product thus separated was filtered, washed with sodium bicarbonate solution (10%) and finally with water. It is then crystallised from ethanol acetic acid mixture to get 1-(5-chloro-2-hydroxyphenyl)-3-(2-chlorophenyl) propane-1, 3-dione [L1]



1-(5-chloro-2-hydroxyphenyl)-3-(2-chlorophenyl)propane-1,3-dione

Synthesis of - 1-(5-chloro-2-hydroxyphenyl)-3-(4-nitrophenyl) propane-1,3-dione [L2]-

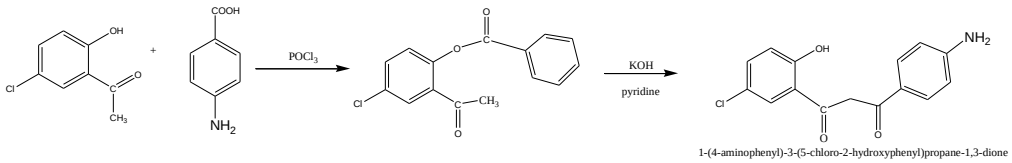
p-nitro substituted benzoyloxy acetophenones prepaired from 4-chloro-2-hydroxy acetophenone (1) (0.04 mol) and chloro substituted benzoic acid (0.05 mol) was dissolved in dry pyridine (40 ml). The solution was warmed up to about 60^{0c} and pulverised KOH (0.15 mol) was added slowly with constant stirring. After four hours the reaction mixture was acidified by adding ice cold dilute HCl (1:1). The solid product thus separated was filtered, washed with sodium bicarbonate solution (10%) and finally with water. It is then crystallised from ethanol acetic acid mixture to get 1-(5-chloro-2-hydroxyphenyl)-3-(4-nitrophenyl) propane-1,3-dione [L2]



1-(5-chloro-2-hydroxyphenyl)-3-(4-nitrophenyl)propane-1,3-dione

Synthesis of - 1-(4-aminophenyl)-3-(5-chloro-2-hydroxyphenyl) propane-1,3-dione [L3]-

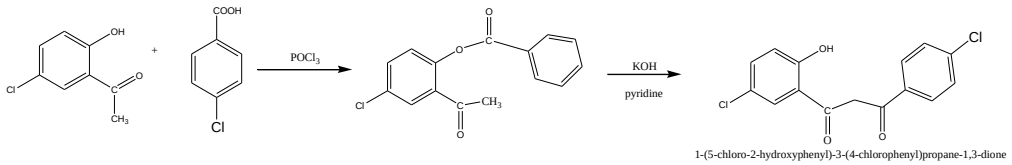
p-amino substituted benzoyloxy acetophenones prepaired from 4-chloro-2-hydroxy acetophenone (1) (0.04 mol) and chloro substituted benzoic acid (0.05 mol) was dissolved in dry pyridine (40 ml). The solution was warmed up to about 60^{0c} and pulverised KOH (0.15 mol) was added slowly with constant stirring. After four hours the reaction mixture was acidified by adding ice cold dilute HCl (1:1). The solid product thus separated was filtered, washed with sodium bicarbonate solution (10%) and finally with water. It is then crystallised from ethanol acetic acid mixture to get 1-(4-aminophenyl)-3-(5-chloro-2-hydroxyphenyl) propane-1,3-dione [L3]



1-(4-aminophenyl)-3-(5-chloro-2-hydroxyphenyl)propane-1,3-dione

Synthesis of - 1-(5-chloro-2-hydroxyphenyl)-3-(4-chlorophenyl) propane-1,3-dione [L4]-

p-chloro substituted benzoyloxy acetophenones prepaired from 4-chloro-2-hydroxy acetophenone (1) (0.04 mol) and chloro substituted benzoic acid (0.05 mol) was dissolved in dry pyridine (40 ml). The solution was warmed up to about 60^{0c} and pulverised KOH (0.15 mol) was added slowly with constant stirring. After four hours the reaction mixture was acidified by adding ice cold dilute HCl (1:1). The solid product thus separated was filtered, washed with sodium bicarbonate solution (10%) and finally with water. It is then crystallised from ethanol acetic acid mixture to get 1-(5-chloro-2-hydroxyphenyl)-3-(4-chlorophenyl) propane-1,3-dione [L4]



Synthesis of - 1-(5-chloro-2-hydroxyphenyl)-3-(2-nitrophenyl) propane-1,3-dione [L5]-
P-chloro substituted benzoyloxy acetophenones prepaired from 4-chloro-2-hydroxy acetophenone (1) (0.04 mol) and chloro substituted benzoic acid (0.05 mol) was dissolved in dry pyridine (40 ml). The solution was warmed up to about 60^{0c} and pulverised KOH (0.15 mol) was added slowly with constant stirring. After four hours the reaction mixture was acidified by adding ice cold dilute HCl (1:1). The solid product thus separated was filtered, washed with sodium bicarbonate solution (10%) and finally with water. It is then crystallised from ethanol acetic acid mixture to get 1-(5-chloro-2-hydroxyphenyl)-3-(2-nitrophenyl) propane-1,3-dione

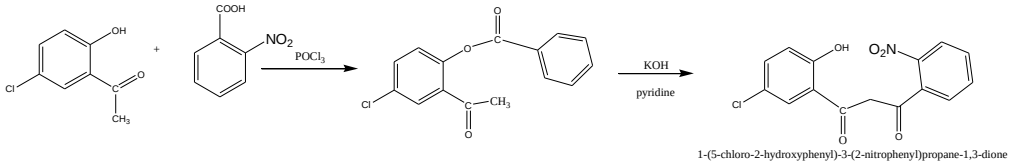


Table-01-Physical data for synthesized compounds [L1-L6]

Comp. Code	Mol. Formula	Mol.Wt. (gms)	Colour	m.p.°C
(L_1)	HC2CP	309	Dark Yellow	125
(L_2)	HC4NP	319.5	Brown yellow	147
(L_3)	HCAP	289.5	Yellow	200
(L_4)	HC4CP	309	Yellow	139
(L_5)	HC2NP	319.5	Brownish	158

Table-02-IR Data Of Substituted diketones

Compound	C=O	-NO_2	-OH	Ph-Cl	Ph-NH_2
(L_1)	1590	---	3429	823.34	---
(L_2)	1691	1345	3067	795.13	---
(L_3)	1599	---	3329	827.32	3477
(L_4)	1588	---	3051	849	---
(L_5)	1595.63	1355	3082.63	826.37	---

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REFERENCES
1. Anto, R. J.; Babu, K. N. D.; Rajasekharan, K. N.; Kuttan, R. Cancer Lett. 1995, 94, 74.
2. Elizabeth K.; Rao, M. N. A. *Int. J. Pharm*. 1990, 58, 237.
3. Antony, S.; Kuttan, R.; Kuttan, G. Immun. Invest. 1999,28, 291.
4. Karvembu, R.; Chinnasamy, Jayabalakrishnan. Trans.Met. Chem. 2000, 27, 574.
5. Kuttan, R.; Bhanumathy, P.; Nirmala, K.; George, M. C.Cancer Lett. 1985, 29, 197.
6. Srimal, R. C.; Dhawan, B. *J. Pharm. Pharmacol*. 1973,25, 447.