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Synthesis and Charecterisation of 2 ,6 -Dichloro-4-[5-(4-Methoxyphynyl) -1-Phynyl-4,5-Dihydro-1h-Pyrazol-3-Yl]Phenol

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Abstract

It was thought interesting to synthesize pyrazole derivatives. The titled compounds were prepared by the action of .Preparation of 1(3,5–dichlaro–4–hydroxylphenyl)–3–(furan–yl)prop–2– en –1–one. With Hydrazine hydrate and phynyl hydrazine. pyrazoles are very useful units in the fields of medicinal and pharmaceutical chemistry and have been reported to exhibit a broad spectrum of biological activities. Due to this vital role of pyrazole derivatives in biological activities,

Keywords*:* phenyl hydrazine, pyrazole.

Introduction

Pyrazoles are heterocyclic compounds containing an unsaturated five membered ring of three carbon atoms united to two nitrogen atoms in 1,2-position. The partially reduced pyrazoles i.e. dihyrdopyrazoles are called pyrazolines. The ring is quite sTable & always inspired chemist for further studies. Pyrazolines were found to have extensive pharmacological activities[1] viz. anti inflammatory[2] , analgesic, anticancer[3], antidepressant[4] activities. Pyrazolines exhibit antibacterial[5,6] ,antifungal[7] , Anti HIV[8], antiviral[9], insecticidal[10] activities hence have a broad spectrum of biological activities[11]. It has been observed that chalcones are the best starting compounds for the preparation of pyrazole derivatives. Encouraged by the earlier reports, we have designed and synthesized some new chlorosubstitutedpyrazole from chalcones and hydrazine hydrate and phynyl hydrazine . The synthetic routes which furnished the target compounds are shown below along with their IR and NMR data.

Materials and Methods

i) Acetic anhydride
ii) 2,6 dichloro phenol
iii) Anhydrous sodium acetate
iv) Anhydrous $AlCl_3$
v) Anisaldehyde
vi) Pipyridine
vii) Hydrazine hydrate & Phenyl Hydrazine
viii) NaOH, KOH, Ethanol & etc.

S D fine, merk and loba companies chemicals are used in the synthesis of Pyrazoles.

Scheme-I

Synthesis of 2,6 dichloro Phenyl Acetate (Ia).

2,6dichloro phenol (25gm) was mixed with acetic anhydride (30ml) and anhydrous sodium acetate (2.5gm).The mixture was Refluxed about 2hr. It was cooled and poured in cold water . The acetate layer was separated and washed with water several times and finally it was purified by distillation and hence distilled compound (A) was collected at about 240^0 C

Synthesis of 1-(3,5-dichloro-4-hydroxyphenyl)ethanone

Take 2,6 dichloro Phenyl Acetate layer was mixed with anhydrous $AlCl_3$ (1:3) and heated at 120^0 C for 45 min on sand bath . The reaction in mixture was decomposed with ice cold water containing a little HCl to get crude product with constant stirring, A pinkish solid obtained which is compound (B) it can be recrystalised by ethanol.

Synthesis of 1(3,5–dichlaro–4–hydroxylphenyl)–3–(furan–2-yl)prop–2– en –1–one:

Take 3,5 dichloro -4- hydroxyphenyl acetate (0.01 mole.) was dissolve in ethanol (10ml) with Anisaldehyde(0.01mole) & added to the solution 1or 2 drops of pipyridine solution & mixture was heated to boiling to this solution add aq. KOH solution (40%) (10ml) was added drop wise with constant stirring. The mixture was stirred mechanically at room temperature for 30 minutes and kept for overnight and then it was acidified by HCl (50%) solution. The solid product thus separated out was filtered and washed with sodium bicarbonate (10%) followed by water the crude product (c) was recrystallised by ethanol.

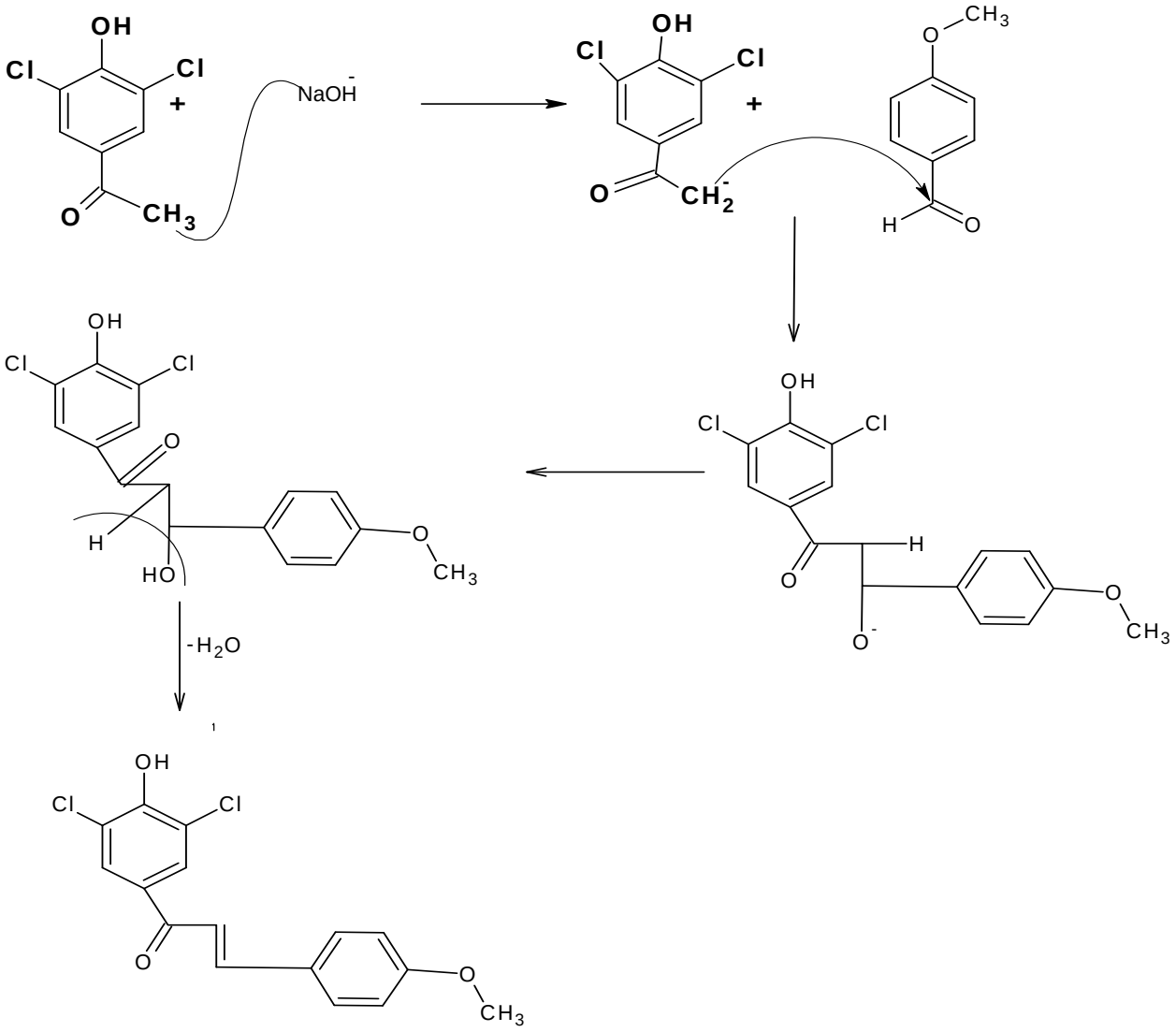
NMR : : δ7.19 (d,-Ar- C-H) , δ6.72(m,-CH_2), δ3.73(s,O-CH_3), IR(KBr ν_{max})(cm^{-1}) 3467cm^{-1}(O-H w),3001cm^{-1} (C=C-O w),2932cm^{-1}(O-H w),1648cm^{-1}(C=C w.).1421cm^{-1}(C=N w.), 1219cm^{-1}(C-O srt.)

Synthesis of 2,6-dichloro-4-[5-(4-methoxyphenyl)-4,5-dihydro-1H-pyrazol-3-yl]phenol

1(3,5–dichlaro–4–hydroxylphenyl)–3–(furan–2-yl)prop–2– en –1–one Chalcone and Hydrazine hydrate mixed in 1:1 ratio were dissolve in ethanol (20ml) to this aqeous KOH solution (0.02m) (10ml) was added the reaction mixture was refluxed for three hours . Cooled diluted with water and acidified with conc. HCl the product thus separated was filterered and recrystalised by ethanol to get compound

NMR: δ6.72 (m,-CH_2) , δ7.01-7.3(d,Ar-C-H), δ3.73(s,O-CH_3), IR(KBr ν_{max})(cm^{-1}) 3076cm^{-1}(-C=C-H w),2662cm^{-1} (O-H w),1609cm^{-1}(C=N srt),1589cm^{-1}(C=C w.).1399cm^{-1}(C-H srt.), 2715cm^{-1}(N-C=O broad)

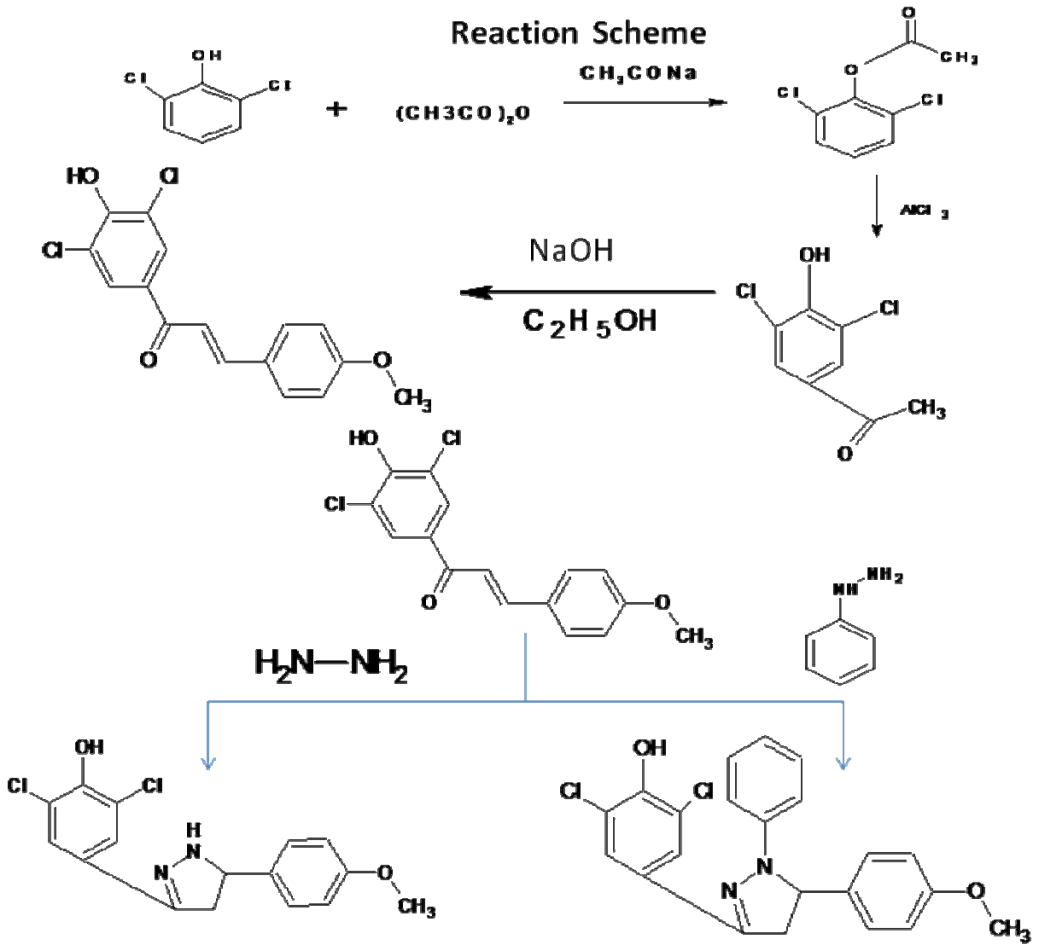
Mechanism:-



Peparation of 2,6-dichloro-4-[5-(4-methoxyphenyl)-1-phenyl-4,5-dihydro-1*H*-pyrazol-3-yl]phenol

2,6-dichloro-4-[5-(4-methoxyphenyl)-1-phenyl-4,5-dihydro-1*H*-pyrazol-3-yl]phenol Chalcone & phenyl hydrazine is gives in 1:1ratio and add 20ml ethanol to this add aq. KOH solution of (0.02m) 10ml was added the reaction mixture was refluxed for three hours cooled diluted with water and acidified with conc. HCl the product thus sepetated was filtered and recrystalised by ethanol to get compound.

IR(KBr ν_{max})(cm^{-1}) 3465cm^{-1}(O-Hstr),3208cm^{-1} (N-H srt.),3003cm^{-1}(H-C=Osrt),1586cm^{-1}(C=Cstr),1607cm^{-1} NMR:δ-5.0(s,O-H),δ7.0-7.6(m,2H,Ar-C-H),δ6.43(d,1H,-CH-),δ2.0(d,CH_2-CH) ,



Result & Discussion:-

Experimental Physical characterization data of all the compounds are given in Table-1.

TABLE 1 Characterization Data of Newly Synthesized Compounds

Compound	Mol. Formula	M.P.(^{0}C)	Yield(%)	R_f
a	$C_8H_6O_2Cl_2$	240 (B.P.)	85	0.75
b	$C_8 H_6 Cl_2O_2$	106^{0}C	75	0.80
c	$C_{13} H_8Cl_2O_3$	152^{0}C	70	0.82
d	$C_{12} H_8 Cl_2 N_2 O_2$	158^{0}C	62	0.89
e	$C_{18} H_{13} Cl_2 N_2 O_2$	164^{0}C	52	0.88

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