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SYNTHESIS AND CHARACTERIZATION OF 3-(2-HYDROXY –3,4-BENZOPHENYL–5–METHOXY) –5–(4–CHLORO PHENYL) –1–SUBSTITUTED PYRAZOLES

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ABSTRACT

1-(2-Hydoxy-3,4-benzophenyl-5-methoxy)-3-(4-chlorophenyl)-propan-1,3-dione was refluxed with semicarbazide/thiosemicarbazide/isonicotinic acid hydrazide for about 2 hours in ethanol solvent (25 mL). The reaction mixture was cooled and then diluted with water. The solid obtained was filtered, washed with water and recrystalized from ethanol to obtain titled compounds, which were characterized by IR and NMR spectral data.

Key words: Synthesis, Pyrazoles**.**

INTRODUCTION

Heterocyclic compounds containing nitrogen play an important role in the metabolism of all living cells. Like other N-heterocyclic compounds, pyrazoles exhibit a wide range of biological activities like, antiviral[1], blood pressure lowering[2], antidepressant[3], antiinflammatory[4], antipyretic[5,6] and antioxidant[7,8]. Pyrazoles are also used as sunscreen materials[9], dyestuffs[10], agrochemicals[11,12] etc. Very few pyrazoles and their derivatives are found in living things, since they are very rare in nature[13]

It has been observed that diketones are the best starting compounds for the pyrazole derivatives. Present work deals with the synthesis of some novel pyrazoles and their characterization by elemental analysis, IR and ^{1}H NMR spectra.

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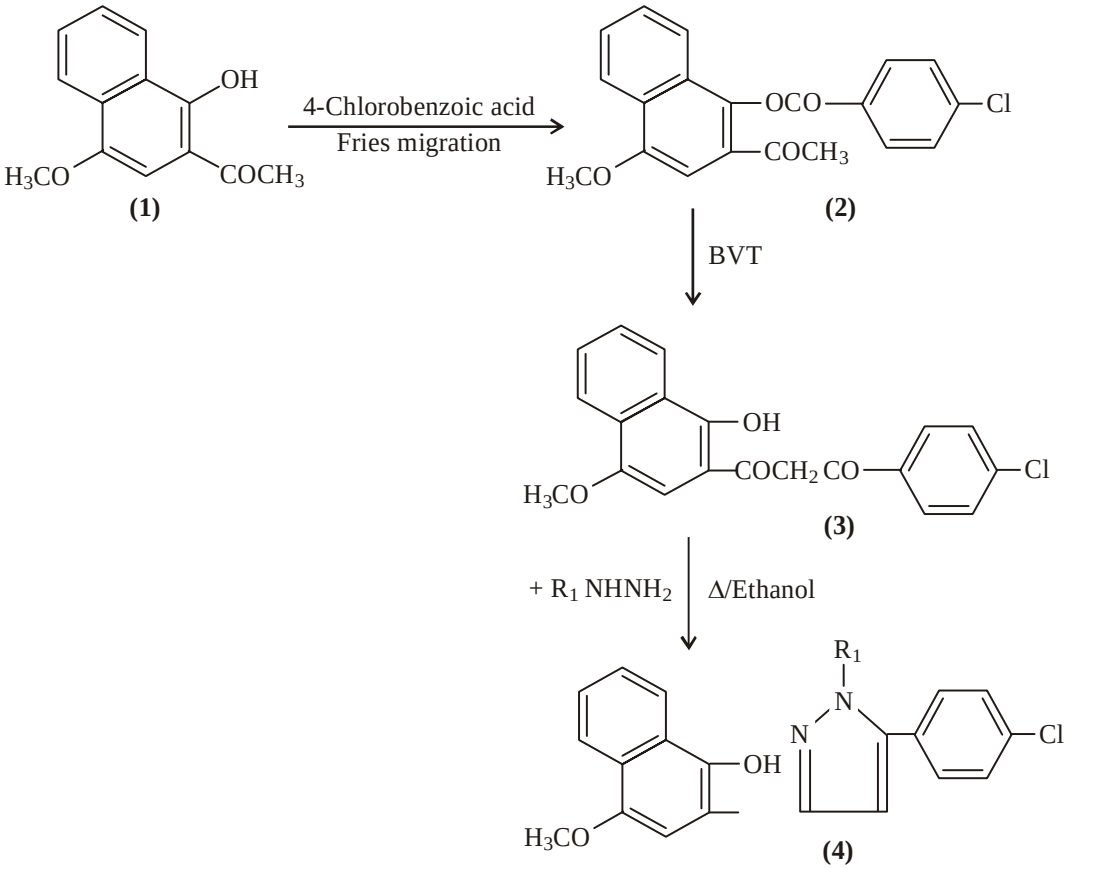
EXPERIMENTAL

All the melting points were taken in silicon oil bath with open capillary tubes and are uncorrected. IR spectra were recorded on a Nicolet-Impact 400 FT-IR spectrometer. ^{1}H NMR spectra were recorded on a Brucker AC300 FNMR spectrometer (300 MHz), using TMS as an internal standard. Microanalysis of nitrogen was obtained by Kjeldahal's method. Thin layer chromatography on silica gel-G, was used to check the purity of the compounds.

Scheme



where $R_1 = CONH_2$ **(4a)**, $R_1 = CSNH_2$ **(4b)**, $R_1 = C_5H_4NCO$ **(4c)**

Synthesis of 2-acetyl-4 methoxy-1-naphthol (1)

In hot glacial acetic acid, fused $ZnCl_2$ was added and refluxed till it dissolved. Then powdered 4-methoxy-1-naphthol was added and refluxed for about 8 hours. The reaction

mixture was cooled and poured in acidulated water. The solid obtained was filtered, washed and recrystallized from ethanol to obtain compound **(1)**. Physical data of the compounds are given in Table 1.

Synthesis of 2-(4-chlorobenzoyloxy)-3,4-benzophenyl-5-methoxyacetophenone (2)

2-Acetyl-4-methoxy-1-naphthol **(1)**, was dissolved in dry pyridine. Reaction mixture containing 4-chloro benzoic acid was treated with $POCl_3$ dropwise with cooling and stirring. During addition of $POCl_3$, the temperature was maintained below 40^oC. The reaction mixture slowly thickened. After 4 to 6 hours, it was treated with dil HCl (1 : 1) The solid obtained was filtered, washed with water and then with $NaHCO_3$ (10%) to remove unreacted organic acid. It was further washed with NaOH (1%) to remove unreacted ketone. The product was finally washed with water and recrystallized from ethanol to obtain compound **(2)**. Physical data of the compound are given in Table 1.

Synthesis of 1-(2-hydroxy-5-methoxy-3,4-benzophenyl-)-3-(4-chlorophenyl)-propan-1,3-dione (3)

2-(4-Chlorobenzoyloxy)-3,4-benzophenyl-5-methoxyacetophenone **(2)** was dissolved in pyridine and treated with pulverized KOH. After 6 hours, the mixture was decomposed with ice cold acetic acid. The crude solid obtained was washed with water followed by $NaHCO_3$ (10%) and finally again with water. The product was filtered and recrystalized from ethanol-acetic acid mixture to obtain compound **(3)**. The physical data are given in Table 1.

Synthesis of 3-(2-hydroxy-3,4-benzophenyl-5-methoxy)-5-(4-chlorophenyl)-1-substituted pyrazoles (4a-c)

1-(2-Hydroxy-5-methoxy-3,4-benzophenyl-)-3-(4-chlorophenyl)-propan-1,3-dione **(3)** was refluxed with semicarbazide / thiosemicarbazide / isonicotinic acid hydrazide for about 2 hours in ethanol solvent . The reaction mixture was cooled and then diluted with water. The solid obtained was filtered, washed with water and recrystallized from ethanol to obtain compounds **(4a-c)**. Their physical data are given in Table 1.

Spectral interpretation of (4a)

IR (ν_{max}) (cm^{-1}): 3403 (OH, str), 3215 (NH_2, str), 1555 (C=N, str) 1706 (CO, str).

NMR (δ ppm): 3.86 (s, 3H, OCH_3), 6.45 (s, 1H, = CH of pyrazole), 6.65-7.38 (m, 9H, Ar-H), 7. 88 (s, 2H, -NH_2), 11.73 (s, 1H, OH).

Table 1: Physical data of synthesized compounds

Compd.	R_1	Melting point (^oC)	% Yield	% Nitrogen		R_f value
				Found	Calculated	
1	--	99	65	--	--	--
2	--	129	62	--	--	--
3	--	149	58	--	--	--
4a	$CONH_2$	203	48	10.52	10.67	0.56
4b	$CSNH_2$	169	45	10.20	10.26	0.58
4c	C_5H_4NCO	172	39	9.18	9.22	0.66

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