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Synthesis of 1,1-Bis-[2-Hydroxy-3-(1′-Benzoyl/Pyridoyl-5′-Aryl-Pyrazolin-3′-yl)-5-Methyl Phenyl] Methane

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New 1,1 bis-[2-hydroxy-3-(1′-benzoyl-5′-aryl-pyrazolin-3′-yl)-5-methyl phenyl] methane (**6a–f**) and 1,1-bis-[2-hydroxy-3-(1′-pyridoyl-5′-aryl-pyrazolin-3′-yl)-5-methyl phenyl] methane (**7a–f**) have been synthesised by the action of 1,1-bis-[2-hydroxy-3-(3-aryl-prop-2-en-1-one)-5-methyl phenyl] methane (**4a–f**) with benzoic acid hydrazide and isoniazid respectively in pyridine medium.

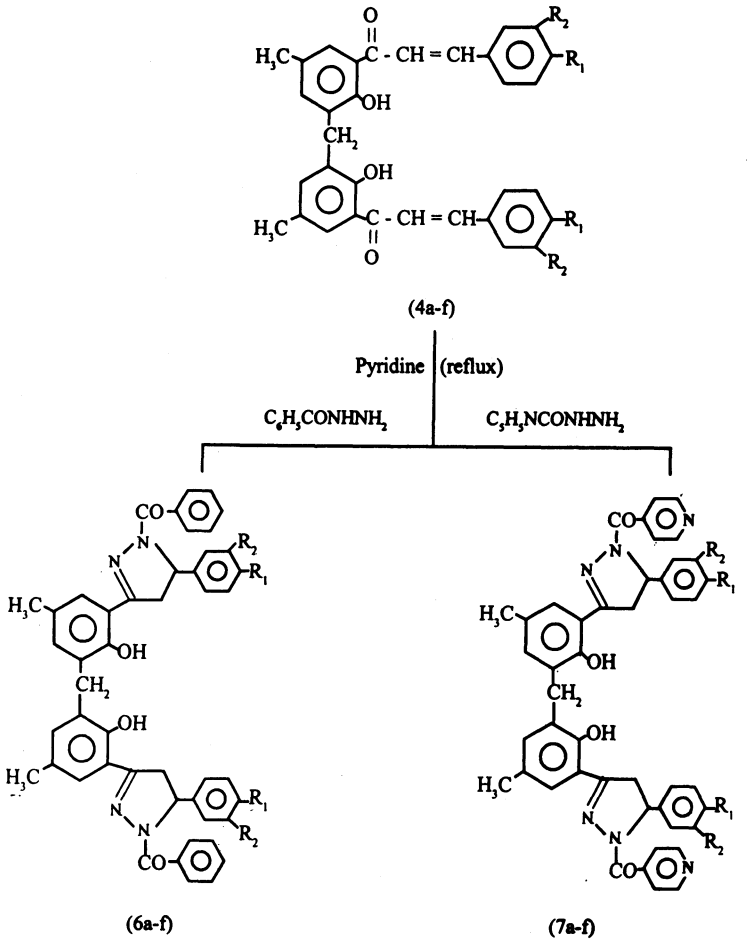
INTRODUCTION

Several pyrazolines are important as pharmaceuticals; they have been found to possess analgesic[1], antipyretic[2], anti-inflammatory[3] and antimicrobial properties[4]. They are also useful as biodegradable agrochemicals[5, 6] and as intermediates in the dye industry[7]. The common method for the synthesis of pyrazolines[8] is one that involves the reaction of hydrazine or phenyl hydrazine on chalcones and flavanones in different solvents like acetic acid, pyridine, ethanol, DMSO etc. Although some bis-pyrazolines are reported[9, 10], but it was observed from literature survey that synthesis of titled bis-pyrazolines from bis-chalcones has not yet been reported.

The present work deals with the synthesis of 1,1-bis-[2-hydroxy-3-(1′-benzoyl-5′-aryl-pyrazolin-3′-yl)-5-methyl phenyl] methane (**6a–f**) and 1,1-bis-[2-hydroxy-3-(1′-pyridol-5′-aryl-pyrazolin-3′-yl)-5-methyl phenyl] methane (**7a–f**) from the reaction of benzoic acid hydrazide and isoniazid with 1,1-bis-[2-hydroxy-3-(3′-aryl-prop-2-en-1-one)-5-methyl phenyl] methane (**4a–f**) respectively in pyridine as a medium. The structures of these compounds have been established on the basis of elemental analysis and spectral analysis (UV, IR, NMR).

EXPERIMENTAL

All melting points were taken in silicon oil bath instrument in open capillary and are uncorrected. Purity of the compounds was checked by TLC on silica gel-G; IR-spectra were recorded on Perkin-Elmer spectrophotometer, PMR spectra on Brucker AC 300 F NMR spectrophotometer at 300 MHz and UV-spectra on Shimadzu spectrophotometer.



Scheme

Preparation of 1,1-bis-[2-hydroxy-3-(1′-benzoyl-5′-aryl-pyrazolin-3′-yl)-5-methyl phenyl] methane (6a–f)

1,1-Bis-[2-hydroxy-3-(3-aryl-prop-2-en-1-one)-5-methyl phenyl] methane **(4a–f)** (0.01 M) was refluxed with benzoic acid hydrazide (0.04 M) for 5–6 h in pyridine solvent. The reaction mixture was decomposed by acidified water. The product obtained was filtered, washed with sufficient water and crystallised from acetic acid.

Spectral interpretation of (6a)

IR (ν_{max}) (cm^{-1}): 3425 ν(OH), 2921 ν(C—H), 1611 ν(C=N), 1470 ν(C=C) and 1252 ν(C—O).

PMR ($CDCl_3$) δ ppm: 2.1 (S, 6H, —CH_3), 3.5 (S, 2H, —CH_2), 3.7 (S, 2H, —OH), 3.9 (d, 4H, —CHH), 5.1 (d, 2H, —CH), 6.6–7.5 (m, 24H, Ar—H)

UV (λ_{max}): 362.3 nm ($n \rightarrow \pi^*$)

Preparation of 1,1-bis-[2-hydroxy-3-(1′-pyridoyl-5′-aryl-pyrazolin-3′-yl)-5-methyl phenyl] methane (7a–f)

1,1-Bis-[2-hydroxy-3-(3-aryl-prop-2-en-1-one)-5-methyl phenyl] methane **(4a–f)** (0.01 M) was refluxed with isoniazid (0.04 M) for 5 h in pyridine solvent. The reaction mixture was decomposed by acidified water. The product obtained was filtered, washed with sufficient water and crystallised from acetic acid.

Spectral interpretation of (7c)

IR (ν_{max}) (cm^{-1}): 3370 ν(OH), 2831 ν(C—H), 1620 ν(C=N), 1469 ν(C=C), 1256 ν(C—O)

PMR ($CDCl_3$) δ ppm: 2.2 (S, 6H, —CH_3), 2.5 (S, 6H, —OCH_3), 3.9 (S, 2H, —CH_2), 4.2 (S, 2H, —OH), 4.3 (d, 4H, —CHH), 5.3 (d, 2H, —CH), 6.8–7.3 (m, 18H, Ar—H)

UV (λ_{max}): 361.8 nm ($n \rightarrow \pi^*$)

TABLE-1
PHYSICAL DATA OF SYNTHESIED BIS-PYRAZOLINES

Compds	R1	R2	m.p. (°C)	Yield (%)	m.f.	N% Found (Calcd.)
6a	—H	—H	223–225	72	$C_{47}H_{40}N_4O_4$	7.82 (7.72)
6b	—OCH_3	—H	215–217	78	$C_{49}H_{44}N_4O_6$	7.21 (7.13)
6c	—OH	—OCH_3	152–154	82	$C_{49}H_{44}N_4O_8$	6.80 (6.86)
6d	—NO_2	—H	> 270	85	$C_{47}H_{38}N_8O_6$	9.92 (10.73)
6e	—H	—OCH_3	210–212	75	$C_{47}H_{44}N_4O_6$	6.98 (7.13)
6f	—NH_2	—H	196–198	72	$C_{47}H_{42}N_6O_4$	12.08 (11.16)
7a	—H	—H	208–210	70	$C_{45}H_{38}N_6O_4$	11.52 (11.56)
7b	—OCH_3	—H	226–228	76	$C_{47}H_{42}N_6O_6$	10.20 (10.68)
7c	—OH	—OCH_3	194–196	85	$C_{47}H_{42}N_6O_8$	10.62 (10.26)
7d	—NO_2	—H	> 270	85	$C_{45}H_{38}N_8O_8$	12.91 (14.27)
7e	—H	—OCH_3	204–206	70	$C_{47}H_{42}N_6O_6$	10.48 (10.68)
7f	—NH_2	—H	191–193	72	$C_{45}H_{40}N_8O_4$	14.83 (14.80)

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