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Synthesis and Characterization of 4-(4-Arylidene)-5-methyl-2, 4-dihydro-3*H*-pyrazol-3-ones

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Abstract
The synthesis of 5-methyl-4-substituted benzylidene-2,4-dihydro-3*H*-pyrazol-3-one (3a-3d) are achieved by the condensation of 5-methyl-2,4-dihydro-3*H*-pyrazol-3-one (1) with 4-substituted benzaldehydes (2a-d). All the synthesized compounds have been analyzed by means of IR, ^{1}H NMR and mass spectral data.

Keywords
Benzaldehydes: Pyrazole derivatives: Sodium acetate: Phenyl: Ethanol

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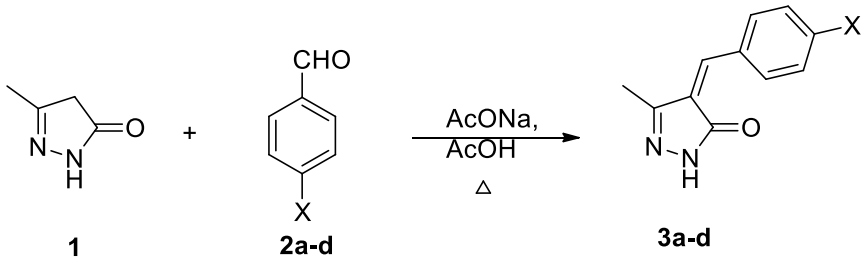
INTRODUCTION:
It was observed from the literature that certain five-member heterocyclic compounds possess interesting biological activity. Among those, pyrazole and their fused derivatives are known to exhibit diverse biological activities and important applications in pharmaceutical industries. Pyrazole derivatives are having potent biological acivities such as, anti pyretic [1], antimicrobial [2], antiviral [3,4], antidepressant [5], antilukemic [6], anti-inflammatory [7], antihistaminic [8], antitumor and anticonvulsant [9]. In view of the pharmaceutical importance of pyrazole moieties, In view of the biological importance of pyrazoles, herein I present synthesis of title compounds.

RESULTS AND DISCUSSION
Synthesis of 4-(4-Arylidene)-5-methyl-2,4-dihydro-3*H*-pyrazol-3-ones was achieved by reaction of 5-Methyl-2,4-dihydro-3*H*-pyrazol-3-one, *p*-chloro-benzaldehyde, and anhydrous sodium acetate at reflux conditions.

As a representative case, the spectral analysis of 4-(4-chlorobenzylidene)-5-methyl-2,4-dihydro-3*H*-pyrazol-3-one **(3a)** is discussed below.

The IR spectrum of compound **3a**, showed the absorption band at about 1722 cm^{-1} and 1610 cm^{-1}corresponding to C=O and C=N stretching. The ^{1}H NMR spectrum of compound 3**a** showed a singlet at δ 8.12 due to methylene (-NH-) group, a multiplet peak at δ 7.08 and 6.18 corresponding to 4 protons of phenyl and pyrazole rings respectively. singlet at δ 1.86 represents CH3 protons.

Scheme-1.



Experimental Section:

The IR spectra were recorded on Perkin-Elmer spectrometer. The ^{1}H NMR spectra were scanned on a Bruker DRX-300 MHz. spectrometer (300 MHz) in $CDCl_3$ using TMS as internal standard and chemical shifts are expressed in δ ppm. The mass spectra were recorded on a Jeol SX-102 (FAB) spectrometer.

Synthesis of 4-(4-chlorobenzylidene)-5-methyl-2,4-dihydro-3*H*-pyrazol-3-one (3a):

5-Methyl-2,4-dihydro-3*H*-pyrazol-3-one (0.01mole), *p*-chloro-benzaldehyde (0.01 mole) and anhydrous sodium acetate (0.02 mole) were dissolved in acetic acid and refluxed for 10 h. The reaction mixture was filtered, and the filtrate was poured on crushed ice. The solid obtained was recrystallized from ethanol.

IR (KBr): 3410 (N-H str.), 3082 (C-H str, Ar-H), 2958 (C-H str, CH_3), 1722 (C=O str.), 1610 (C=N str.), 732 (C-Cl str.) cm^{-1}.; ^{1}H NMR ($CDCl_3$): δ 8.12 (s, 1H, NH), 7.08 - 7.69 (m, 4H, Ar-H), 6.18 (s, 1H, =CH-Ar), 1.86 (s, 3H, CH_3).

4-(4-Methoxybenzylidene)-5-methyl-2,4-dihydro-3*H*-pyrazol-3-one (3b): IR (KBr): 3439 (N-H str.), 3081 (C-H str, Ar-H), 2959 (C-H str, CH_3), 1686 (C=O str.), 1606 (C=N str.), 1095 (C-O str.) cm^{-1}.; ^{1}H NMR ($CDCl_3$): δ 8.20 (s, 1H, NH), 7.14 - 7.50 (m, 4H, Ar-H), 6.08 (s, 1H, = CH-Ar), 3.73 (s, 3H, OCH_3), 1.70 (s, 3H, CH_3).

4-(4-*N,N*-Dimethylaminobenzylidene)-5-methyl-2,4-dihydro-3*H*-pyrazol-3-one (3c): IR (KBr): 3412 (N-H str.), 3070 (C-H str, Ar-H), 2953 (C-H str, CH_3), 1682 (C=O str.), 1604 (C=N str.) cm^{-1}.; ^{1}H NMR ($CDCl_3$): δ 8.25 (s, 1H, NH), 7.31 - 7.66 (m, 4H, Ar-H), 6.11 (s, 1H, = CH-Ar), 3.29 (s, 6H, $N(CH_3)_2$), 1.93 (s, 3H, CH_3).

4-Benzylidene-5-methyl-2,4-dihydro-3*H*-pyrazol-3-one (3d): IR (KBr): 3392 (N-H str.), 3066 (C-H str, Ar-H), 2949 (C-H str, CH_3), 1708 (C=O str.), 1595 (C=N str.) cm^{-1}.; ^{1}H NMR ($CDCl_3$): δ 8.16 (s, 1H, NH), 7.18 - 7.63 (m, 5H, Ar-H), 6.36 (s, 1H, =CH-Ar), 1.83 (s, 3H, CH_3).

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