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**Der Pharma Chemica, 2010, 2(1): 168-172**
*(http://derpharmachemica.com/archive.html)*

**ISSN 0975-413X**

# Synthesis, characterization and antimicrobial activities of 1-Acetyl-5-(substituted phenyl)-{3-[4-(2-methyl-4-benzylidene-5-oxo-imidazol-1-yl)]phenyl}-4,5-dihydropyrazol

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**Abstract**

1-acetyl-5-(substituted phenyl)-{3-[4-(2-methyl-4-benzylidene-5-oxo-imidazol-1-yl)]phenyl} -4,5-dihydropyrazol have been prepared by the refluxation for three hours of 5-(substituted phenyl)-{3- [4- (2-methyl- 4-benzylidene-5-oxo-imidazol-1-yl)]phenyl}-4,5-dihydropyrazol and acetic acid. the intermediate P-3 have been prepared by the refluxation for three hours of 4-benzylidene-1-{4-[3-(substituted phenyl)prop-2-enoyl] phenyl} -2 –methyl -imidazol-5-one with hydrazine hydrate in presence of ethanol the intermediate P-2 synthesized by the condensation of 1-(4-acetylphenyl)-4-benzylidene-2-methyl-imidazol-5-one with various aldehydes.

**Key Words :** pyrazolines, hydrazinehydrate, benzaldehyde , oxazolone, pyrazol.

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## Introduction

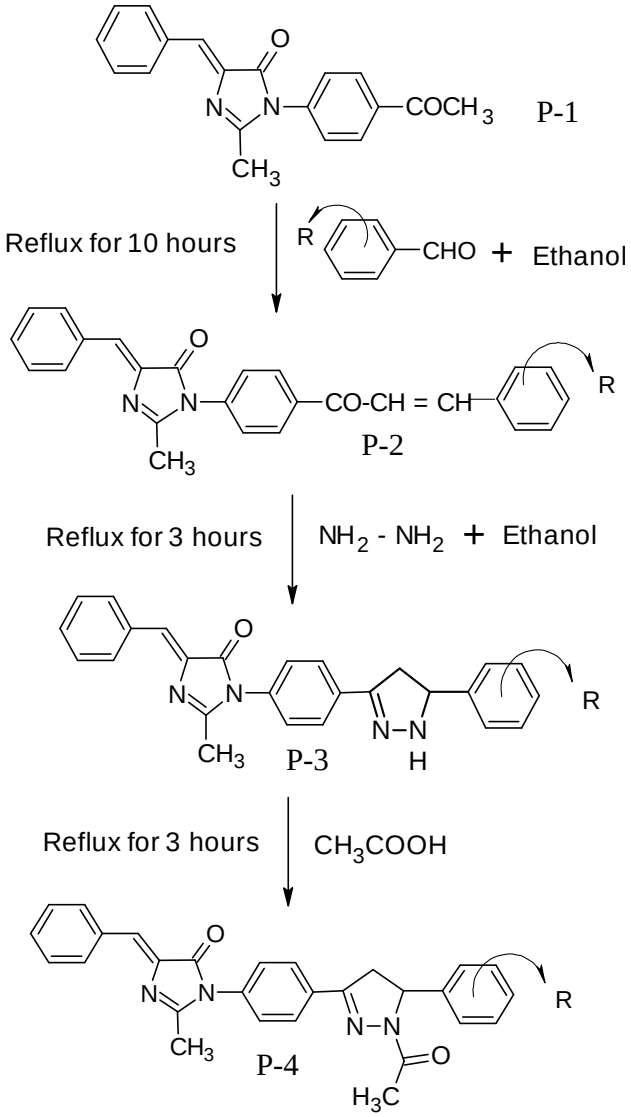
In view of our on going interest in the synthesis of nitrogen containing heterocyclic, the synthesis of biologically active pyrazolines has been undertaken. Pyrazolines derivatives possess wide range of biological and physiological activities such as antiimplantation(1), antitumor(2), antiarthritic(3), analgasic(4), anti-inflammatory (5), and industrial applications(6). To further assess the anticancer and antitubercular activities of pyrazolines, we have synthesized the pyrazoline derivatives reported herein. All the compounds have been prepared by known literature methods(7).

## Experimental

Melting points were taken in open capillary tube and were uncorrected. IR spectra (KBr) were recorded on I.R. Spectrophotometer of Buck scientific Model No. 500 and instrument used for NMR Spectroscopy was DUL 13C-1, 300 MHz and tetramethyl silane used as internal standard. Solvent used were $CDCl_3$ and DMSO. Purity of the compounds were checked by tlc on silica- G plates. Anti microbial activities were tested by Cup-Borer method.

Standard drugs like Penicillin, Kanamycine, Baycor 25 w.p and Amphotericin B were used for the comparison purpose (Table-2)

# Reaction Scheme




**Preparation of *1-(4-acetylphenyl)-4-benzylidene-2-methyl-imidazol-5-one* (P-1).**

In a 250 ml conical flask equipped with a reflux condenser a mixture of 4-benzylidene-2-methyl-1,3-oxazol-5-one(18.719g, 0.1M), 1-(4-aminophenyl)ethanone (13.51g, 0.1M), 25 ml pyridine and about one pellet of KOH was placed and was heated on sand bath for 7-8 hours. Then the mixture was poured in ice. The precipitates were collected, washed with 10% HCl and re-crystallized from ethanol. The yield of the product was 72 % and the product melts at 112 $^0$C.

Found: C(74.94%) H(5.26%) N(9.18%) , Calcd. for $C_{19}H_{16}N_2O_2$: C(74.98%) H(5.30%) N(9.20%), IR (KBr); (cm$^{-1}$): 3080(= CH-), 2960(-CH Stretch), 1705(>C=O imidazolone), 1650(>C=N-), 1600(>C = C<), 1375(-$CH_3$bend), 1260(C-N).

**Preparation of *4-benzylidene-1-{4-[3-(substituted phenyl)prop-2-enoyl]phenyl}-2-methyl-imidazol-5-one* (P-2).**

The solution of 1-(4-acetylphenyl)-4-benzylidene-2-methyl-imidazol-5-one(3.043g, 0.01M) in absolute ethanol (50 ml),substituted benzaldehyde (0.01M) and 2% NaOH (10 ml) were added and reflixed for 10 hours. After refluxing the reaction mixture was concentrated , cooled, filtered and neutralized with dil. HCl. The solid residue thus obtained was recrystallized with suitable solvent.

IR (KBr);P-2a ($cm^{-1}$)**:** 3080(= CH-), 2900(-CH Stretch), 1725(>C=O imidazolone), 1675(>C=N-), 1590(>C = C<), 1375(-$CH_3$bend), 1260 (C-N), 700(C-Cl), NMR ;P-2f: 2.501, singlate (3H)(-$CH_3$), 3.490, singlate (3H)(-$OCH_3$), 5.631 , singlate (1H) (=CH-vinylic), 6.660-7.902 , multiplate (14H) ( Ar-H) 8.262, singlate (1H)(-OH)

**Table:1 Physical constant of 1-acetyl-5-(substitutedphenyl)-{3-[4-(2-methyl-4-benzylidene -5-oxo-imidazol-1-yl)]phenyl}-4,5-dihydropyrazol**

| No. | Sub. No. | R | Molecular Formula | Mol. Wt. (g/m) | Yield (%) | M. P. $^0$C | Carbon (%) | | Hydrogen (%) | | Nitrogen (%) | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | Found | required | Found | required | Found | required |
| 1 | P-4a | -4-Cl | $C_{28}H_{23}ClN_4O_2$ | 482.96082 | 63 | 210 | 69.61 | 69.63 | 4.78 | 4.80 | 11.57 | 11.60 |
| 2 | P-4b | -2-Cl | $C_{28}H_{23}ClN_4O_2$ | 482.96082 | 68 | 205 | 69.60 | 69.63 | 4.79 | 4.80 | 11.56 | 11.60 |
| 3 | P-4c | -3-$OCH_3$,-4-$OCH_3$ | $C_{30}H_{28}N_4O_4$ | 508.56772 | 68 | 183 | 70.83 | 70.85 | 5.51 | 5.55 | 11.00 | 11.02 |
| 4 | P-4d | -2-$NO_2$ | $C_{28}H_{23}N_5O_4$ | 493.51332 | 59 | 188 | 68.12 | 68.14 | 4.67 | 4.70 | 14.17 | 14.19 |
| 5 | P-4e | -2-OH | $C_{28}H_{24}N_4O_3$ | 464.51516 | 62 | 181 | 72.39 | 72.40 | 5.20 | 5.21 | 12.03 | 12.06 |
| 6 | P-4f | -3-$OCH_3$,-4-OH | $C_{29}H_{26}N_4O_4$ | 494.54114 | 72 | 211 | 70.41 | 70.43 | 5.28 | 5.30 | 11.31 | 11.33 |
| 7 | P-4g | -4-OH | $C_{28}H_{24}N_4O_3$ | 464.51516 | 75 | 231 | 72.37 | 72.40 | 5.20 | 5.21 | 12.03 | 12.06 |
| 8 | P-4h | -4-$N(CH_3)_2$ | $C_{30}H_{29}N_5O_2$ | 491.58356 | 70 | 240 | 73.29 | 73.30 | 5.93 | 5.95 | 14.22 | 14.25 |
| 9 | P-4i | -4-$OCH_3$ | $C_{29}H_{26}N_4O_3$ | 478.54174 | 66 | 232 | 72.76 | 72.79 | 5.45 | 5.48 | 11.70 | 11.71 |
| 10 | P-4j | -3-$OCH_3$,-4-$OCH_3$,-5-$OCH_3$ | $C_{31}H_{30}N_4O_5$ | 538.5937 | 75 | 246 | 69.11 | 69.13 | 5.60 | 5.61 | 10.38 | 10.40 |

**Preparation of *5-(substituted phenyl)-{3-[4-(2-methyl-4-benzylidene-5-oxo-imidazol-1-yl)]phenyl}-4,5-dihydropyrazol* (P-3).**

A mixture of 4-benzylidene-1-{4-[3-(substituted phenyl)prop-2-enoyl]phenyl}-2-methyl-imidazol-5-one(0.01M) and 99% hydrazine hydrate (0.015M) in ethanol (50ml) was refluxed gently for 3 hours. Then the mixture was concentrated and allowed to cool. The resulting solid was filtered, washed with ethanol and recrystallised from ethanol to give a pale brown solid.

IR (KBr);P-3a: ($cm^{-1}$):3350 (>N-H), 3080 (= CH-) , 2910 (-CH),1725 (>C=O imidazolone), 1645 (>C=N-), 1570(>C = C<), 1450 (>$CH_2$ pyrazoline), 1375 (-$CH_3$ bend) , 1250 (N-N), 1150 (C-N), 680 (C-Cl); NMR ;P-3j: 0.900 , dublate (2H)(>$CH_2$), 1.216 , triplate (1H) (>CH-), 2.490 , singlate (3H)(-$CH_3$), 3.383 , singlate (9H)(-$OCH_3$), 3.802 , singlate (1H)(-NH-), 5.515 , singlate (1H) (=CH-vinylic), 7.191-7.240 , multiplate (11H) ( Ar-H).

**Preparation of *1-acetyl-5-(substituted phenyl)-{3-[4-(2-methyl-4-benzylidene-5-oxo-imid azol-1-yl)]phenyl}-4,5-dihydropyrazol* (P-4)**

A mixture of 5-(substituted phenyl)-{3-[4-(2-methyl-4-benzylidene-5-oxo-imidazol-1-yl)]phenyl}-4,5-dihydropyrazol(0.001M) and acetic acid (10ml) was refluxed for 3 hours. The solution was then concentrated, on cooling, the resulting solid was filtered, washed with water and recrystallised from ethanol.

IR (KBr);P-4c: ($cm^{-1}$): 3090 (= CH-) , 2910 (-CH),1720 (>C=O imidazolone), 1650 (>C=N-), 1500(>C = C<), 1450 (>$CH_2$ pyrazoline), 1375(-$CH_3$bend), 1250 (C-O), 1280 (N-N), 1150 (C-N); NMR ;P-4h**:** 1.253, dublate (2H)(>$CH_2$), 1.675, triplate (1H) (>CH-), 1.793, singlate (6H)(-N($CH_3$)$_2$), 1.915 , singlate (3H)(-$COCH_3$), 2.902, singlate (3H)(-$CH_3$), 5.565 , singlate (1H) (=CH-vinylic), 7.263 , multiplate (13H) ( Ar-H).

**Table : 2 Antimicrobial activities of 1-acetyl-5-(substitutedphenyl)-{3-[4-(2-methyl-4-benzylidene -5-oxo-imidazol-1-yl)]phenyl}-4,5-dihydropyrazol**

| Sr. No. | Comp. No. | R | Zone of inhibitions in mm | | |
|---|---|---|---|---|---|
| | | | E.coli | S. aureus | C. albicans |
| 1 | P-4a | - 4-Cl | 16 | 15 | 18 |
| 2 | P-4b | - 2-Cl | 13 | 14 | 16 |
| 3 | P-4c | - 3-$OCH_3$, -4-$OCH_3$ | 16 | 14 | 16 |
| 4 | P-4d | - 2-$NO_2$ | 14 | 12 | 13 |
| 5 | P-4e | - 2-OH | 12 | 11 | NA |
| 6 | P-4f | - 3-$OCH_3$, -4-OH | NA | 13 | 14 |
| 7 | P-4g | - 4-OH | 13 | 12 | 12 |
| 8 | P-4h | - 4-N($CH_3$)$_2$ | 12 | 12 | 12 |
| 9 | P-4i | - 4-$OCH_3$ | 11 | 13 | 13 |
| 10 | P-4j | - 3-$OCH_3$, -4-$OCH_3$, -5-$OCH_3$ | 17 | 14 | 19 |
| 11 | Penicillin | - | 18 | 20 | - |
| 12 | Kanamycine | - | 19 | 24 | - |
| 13 | Baycor 25 w.p | - | - | - | 24 |
| 14 | Amphotericine | - | - | - | 21 |

## Acknowledgements

The authors are thankful to the Sheth L.H.Science College, Mansa for providing research facilities. One of the author Pankaj S. Patel is thankful to UGC. Ganeshkhind, pune.for Teacher Research Fellowship.

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