 ISSN: 2321-8169
28 – 30

# Mocrowave Assited Synthesis & Microbial Activities of some Pyrazoline Derivatives

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**Abstract** - Some new pyrazolines3-methyl-1, 4, 5-triphenyl-1, 3a, 4, 5tetrahydropyrazolo[3-4c]pyrazole, 4(4-Bromophenyl) – methyl -1, 5-diphenyl -1, 3a, 4, 5 – tetra hydrophyrozolo [3, 4c] pyrazole, 4(4-chlorophenyl) – 3 methyl -1, 5-diphenyl -1, 3a, 4, 5 – tetrahydrophyrozolo [3, 4c] pyrazole 4(4-Nitrophenyl) – 3 methyl -1, 5-diphenyl -1, 3a, 4, 5 – tetra hydrophyrozolo [3, 4c] pyrazole, 4(4-methoxyphenyl) – 3 methyl -1, 5-diphenyl -1, 3a, 4, 5 – tetra hydrophyrozolo [3, 4c] pyrazole, 4(4 methyl -2, 6-diphenyl -2, 3, 3a, 6 – tetra hydrophyrozolo [3, 4c] pyrazol-3-yl)phenol, pyrazole are prepared by condensation of 1-phenyl-3-methyl-5-pyrazolone with substituted benzaldehyde under microwave irradiation afford chalcones which under cyclization with phenyl hydrazine to afford pyrazolines.
Synthesized compound confirmed by suitable spectroscopic technique such as $^1$HNMR. The compounds were screened for their in antifungal activity against Rhizopus Oryzae and Penicillium notatum.

***Keywords:*** *Microwave irradiation, Pyrazolines, spectral data, antifungal activity.*

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## I. Introduction

The pyrazoline function is quit stable and inspired chemists to utilize this stable fragment on bioactive moieties to synthesize new compounds possessing biological activities.A number of biological activity are associated with pyrazoline[1-3], pyrazoline heterocycles having two nitrogens at [1, 2] position, respectively. Numerous reports have appeared in literature ascribing antimicrobial[4-5], analagesic[6], antipyretic[7], insectidal[8], diuretic[9], and cardiovascular[10] properties of heterocyclic ring such a pyrazoline[11-13]. Nitrogen containing heterocyclic compounds[14] like pyrazolines have received considerable attention in recent years due to their biological activity like anti-inflammatory[15], anticonvulsant[16]. On the other hand, microwave assisted organic reactions have emerged as a new Lead in organic synthesis with important advantages like highly accelerated rate of reaction along with improvement in yield and quality of product10. Thus keeping in view the advantages ofthese techniques and immense biological importance of pyrazolines, it was felt worthwhile to study the reaction under microwave irradiation and to screen the target compounds for antimicrobial activity.

## II. Experimental

All melting points were determined in open capillary tubes and are uncorrected. All the chemicals and solvents used were of laboratory grade. The IR spectra were recorded on a PERKIN ELMER spectrometer in the frequency range 4000 – 400 cm$^{-1}$ in Nujol mull and as KBr pellets. $^1$H NMR spectra was recorded on Brucker 400MHz, NMR spectrometer using TMS as an internal standard. Reactions were carried out in a domestic microwave oven at 180 watt.

### 2. 1 General Method

Firstly synthesized 1-phenyl-3-methyl-5-pyrazolone. Then this 1-phenyl-3-metheyl-5-pyrazolone (0. 05mole) and substituted benzaldehyde (0. 05mole) in glacial acetic acid taken in conical flask sodium acetate was added into reaction micture. Reaction mixture zapped in microwave oven for 1 min to 2 min at 180 watt and then cooled in refrigerator overnight. The product obtained was filtered and washed with water and recrystallization from ethanol. Then these substituted benzylidene pyrazolone (IIIa-IIIe) reacts with phenyl hydrazine in microwae oven for 3 min at 180 watt gives different substituted fused pyrazoline.

### 2. 2 Synthesis of substituted phenyl -3-methyl -1,4,5-triphenyl -1, 3a, 4, 5 tetrahydrapyrazolo [3-4c] pyrazole (VIa-VIe).

A mixture of substituted benzylidene pyrazolone (0. 025) (IIIa-IIIe) reacts with phenyl hydrazine (0. 025) in microwave oven for 1 to 3 min at 180 watt. After cooling the solution was poured in to crushed ice and the product obtained was filtered & recrystallized using ethanol.

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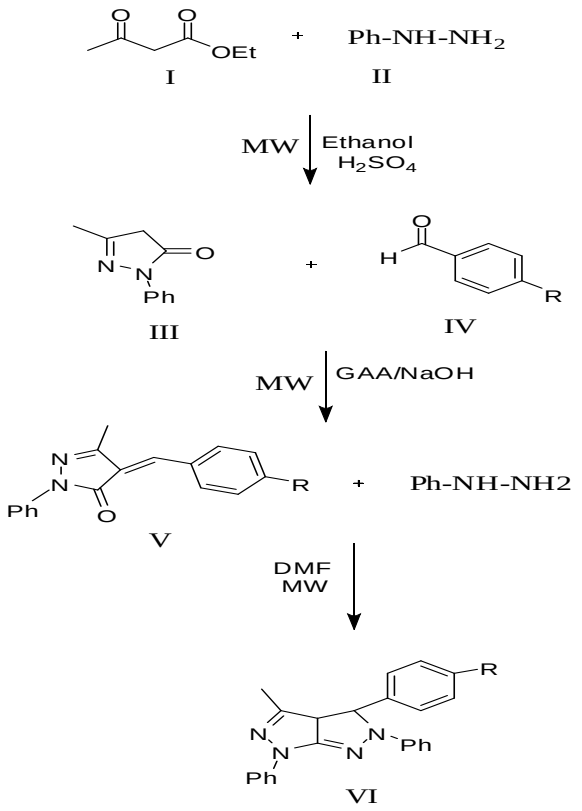


**2. 3 Physical data of synthesized compounds.**

**Table 1**

| Sr. No. | Compound | R | Reaction time (min) | Molecular Formula | Molecular weight | Yield | M. Pt. |
|---|---|---|---|---|---|---|---|
| 1 | VI a | -H | 38 Sec. | $C_{23}H_{20}N_4$ | 352 | 60 | 150 °C |
| 2 | VI b | -Cl | 2 Min | $C_{23}H_{19}N_4Cl$ | 386.5 | 58 | 170 °C |
| 3 | VI c | -Br | 37 Sec. | $C_{23}H_{19}N_4Br$ | 430.9 | 58 | 175 °C |
| 4 | VI d | -$NO_2$ | 37 Sec. | $C_{23}H_{19}N_5O_2$ | 397 | 57 | 185 °C |
| 5 | VI e | -$OCH_3$ | 1.6 Min | $C_{24}H_{22}N_4O$ | 382 | 67 | 170 °C |

**[1]HNMR Spectrum:**

**1) 3 – Methyl 1, 4, 5 triphenyl, 1, 3a, 4, 5 tetrahydropyrazolo [3-4c] pyrazole (VIa)**

1HNMR (400MHZ DMSO, δPPM) 7. 8 (m, Ar-H), 7. 44 (m, Ar-H), 7. 2(m, Ar-H), 1.
96(3H, -CH3), 3. 5(CH, methine) 2. 32 (CH, methine)

**2) 4(4-chlorophenyl) – 3 methyl -1, 5-diphenyl -1, 3a, 4, 5 – tetra hydrophyrozolo [3, 4c] pyrazole (VIb)**

1HNMR (400MHZ DMSO, δPPM) 7. 8 (m, Ar-H), 7. 45 (m, Ar-H)7. 35 (m, Ar-H)7. 35
(m, Ar-H), 7. 47(S, c-cl)2. 33 (3H, methyl), 5(S, C- N)

**3) 4(4-Bromophenyl) – 3 methyl -1, 5-diphenyl -1, 3a, 4, 5 – tetra hydrophyrozolo [3, 4c] pyrazole (VIc)**

1HNMR (400MHZ DMSO, δPPM) 7. 82(m, Ar-H), 8. 0(m, Ar-H), 7. 5(m, Ar-H), 7. 36(S,
C-Br), 5. 32(S, C-N), 3. 41(CH, methine), 6. 73(1-benzene, 1-N)

**4) 4(4-Nitrophenyl) – 3 methyl -1, 5-diphenyl -1, 3a, 4, 5– tetra hydrophyrozolo [3, 4c] pyrazole (VId)**

1HNMR (400MHZ DMSO, δPPM) 8. 1(m, Ar-H), 7. 8(m, Ar-H), 7. 25(m, Ar-H), 7. 54(S,
NO2), 2. 37(CH, methine)

**5) 4(4-methoxyphenyl) – 3 methyl -1, 5-diphenyl -1, 3a, 4, 5 – tetra hydrophyrozolo [3, 4c] pyrazole (Ie)**

1HNMR (400MHZ DMSO, δPPM) 8. 02(m, Ar-H), 7. 81(m, Ar-H), 7. 83(m, Ar-H), 6.97
(S, C-O), 1. 97(S,methyl), 3. 80(CH, methine), 4.95(n-Ar)

## III. Antimicrobial Activities

**3. 1 Antifungal Activity**

Rhizopus Oryzae And Penicillium notatum species were taken for the study of antifungal actity.The antimicrobial activity was determined by using disc diffusion method by measuring the inhibition zone in mm. All synthesized compound exhibited significant antifungal activity.

**Table no 2**

| Compound | Zone of inhibition in mm | |
|---|---|---|
| | Antifungal | |
| | ***R. oryzae*** | ***P. notatum*** |
| VIa | Resistant | 15 |
| VIb | 10 | 12 |
| VIc | 15 | 12 |
| VId | 15 | 15 |
| VIe | 20 | 18 |
| Streptomycin | 16 | 18 |

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## IV. Results and Discussions

Chalcones (IIIa-IIIe) were prepared by followings the standardprotocol (II) and were reacted phenyl hydrazine to yield 4-substituted phenyl-3-methyl, 5-diphenyl–1, 3a, 4, 5–tetrahydropyrazolo [3-4c] pyrazole (VIa-VIe). The synthetic procedure for preparation of compounds is given in scheme I. The assigned structure of newly synthesized compounds (VIa-VIe) were confirmed and supported by $^{1}$HNMR and which was in full agreement with proposed structures. The compounds were screened in vitro antifungal potential by disc diffusion method against pathogenic bacteria. The results of antifungal activities expressed in terms of inhibition zone are reported in Table no. 2. Even though the synthesized compound shows appreciable antibacterial activity.

## V. Conclusion

Few novel pyrazoline derivatives (VIa-VIe) have been synthesized and evaluated for antimicrobial activity. The results of antimicrobial studies of newly synthesized compounds related that they possess significant antibacterial activities.

## VI. Acknowledgment

Authors are thankful to SAIF Chandigarh for recording spectra and to the Head, Department of Microbiology, Shri Shivaji College, Akola.

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