

# Synthesis, Characterization and Microbiological Activity of Schiff Base “1- (2, 4-Dinitrophenyl) -4- (Substitutedphenyl) -6- {[(4-Chloro)phenyl] Methylene} Amino} -3-Methyl-1, 4-Dihydro Pyrano [2, 3-C] Pyrazole-5-Carbonitrile”

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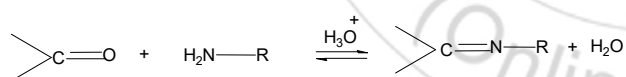
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**Abstract:** A Schiff Base 1- (2, 4-dinitrophenyl) -4- (substitutedphenyl) -6- {[(4-chloro)phenyl] methylene} amino} -3-methyl-1, 4-dihydro pyrano [2, 3-c] pyrazole-5-carbonitrile have been synthesized by reaction between 6-amino-4- (substitutedphenyl) -1- (2, 4-dinitrophenyl) -3-methyl-1, 4-dihydro pyrano [2, 3-c] pyrazole-5-carbonitrile and 4-chlorobenzaldehyde in presence of ethanol the intermediate 6-amino-4- (4-chlorophenyl) -1- (2, 4-dinitrophenyl) -3-methyl-1, 4-dihydro pyrano [2, 3-c] pyrazole-5-carbonitrile was synthesized by the reaction between 2, 4-di-Nitro Phenyl Hydrazine and substituted aldehyde in the presence of malano nitrile, piperidine and ethanol 2- (2, 4dinitrophenyl) -5-methyl-2, 4-dihydro-pyrazol-3-one which also synthesized from of ethyl acetoacetate and 2, 4-di-Nitro Phenyl Hydrazine. The synthesized compounds were characterized by means of their IR, <sup>1</sup>H-NMR spectral data and elemental analysis. All the synthesized products were evaluated for their antimicrobial activities by Cup borer method.

**Keywords:** pyrazol-3-one, aldehyde, Ethenol

## 1. Introduction

A compounds having structure, R-CH = NR<sup>1</sup> or R-C=NR<sup>1</sup> and their related derivatives. are known as imines, azomethines, anils or more commonly as schiff bases (where R&R<sup>1</sup>=alkyl or aryl) [1]. When aldehydes or ketones react with primary amine (R-NH<sub>2</sub>) to form compounds with a carbon – nitrogen double bond they are called imines. The reaction is acid catalyzed. These compound were discovered by Schiff [2] and so they are commonly known as Schiff bases.



Anticonvulsant activity of the schiff base derivatives obtained from vanillin was established by Tiwari S. S., Husain M. I. and Srivastava G. C [3]. Dash B., Patra M. and Mahapatra P., J [4] have synthesized and tested schiff base derivatives for fungicidal activity. Profft Elmar and Hogel Egon., C. A. [5] reported the synthesis and anthelmintic activity of schiff base derivatives. Turan-Zitouni et al. have prepared some 5-Bromoimidazo [1, 2a] pyridine-2-carboxylic acid benzyldenehydrazide and screened their antimicrobial activity [6]. Terzioglu and Gursoy have synthesized some novel 2, 6-dimethyl-N-substituted phenylmethyleneimidazo [2, 1-b] [1, 3, 4] thiadiazole-5-carbohydrazides showed the most favorable cytotoxicity [7]. Trivedi D. K. and Desai K.R., [8] synthesized 1- [4'-imino (substituted phenyl) phenyl] -2-phenyl-4-{4''-N, N-dimethyl amino benzyldene}-imidazol-5-one (d-11) and study of their anti microbial activity. Pawar et al. [9] have synthesized schiff base derivatives and tested for their anti bacterial activity.

## 2. Experimental

### Preparation of 6-amino-4- (substitutedphenyl) -1- (2, 4-dinitrophenyl) -3-methyl-1, 4-dihydro pyrano [2, 3-c] pyrazole-5-carbonitrile (B 1 to 10)

6 - amino- 4- (substitutedphenyl) -1- (2, 4-dinitrophenyl) -3-methyl -1, 4-dihydro pyrano [2, 3-c] pyrazole-5-carbonitrile have been prepared by the refluxation for five hours of 2- (2, 4-dinitrophenyl) -5-methyl-2, 4-dihydro-pyrazol-3-one, malononitrile, substitutedbenzaldehyde and piperidine in presence of ethanol the intermediate 2- (2, 4dinitrophenyl) -5-methyl-2, 4-dihydro-pyrazol-3-one (A) synthesized by the condensation of Ethyl acetoacetate and 2, 4-di-Nitro Phenyl Hydrazine. The synthesized compounds were characterized by means of their IR, <sup>1</sup>H-NMR spectral data and elemental analysis. All the synthesized products were evaluated for their antimicrobial activities by borer method.

### Preparation of 1- (2, 4-dinitrophenyl) -4- (substitutedphenyl) -6- {[(4-chloro)phenyl] methylene} amino} -3-methyl-1, 4-dihydro pyrano [2, 3-c] pyrazole-5-carbonitrile (C 1 to 10)

6-amino-4- (4-chlorophenyl) -1- (2, 4-dinitrophenyl) -3-methyl-1, 4-dihydro pyrano [2, 3-c] pyrazole-5-carbonitrile (0.01M) react with 4-chlorobenzaldehyde (0.01M) and absolute alcohol (30ml) were placed and 1 to 2 drops of hydrochloric acid was added and the mixture was then heated on water bath for 6 hours and then cooled and the precipitates were filtered off and re-crystallized from ethanol melting points were taken in open capillary tube and were uncorrected. IR spectra were recorded on I.R.Spectrophotometer of Bruker scientific Model No. Alpha E and instrument used for NMR Spectroscopy was

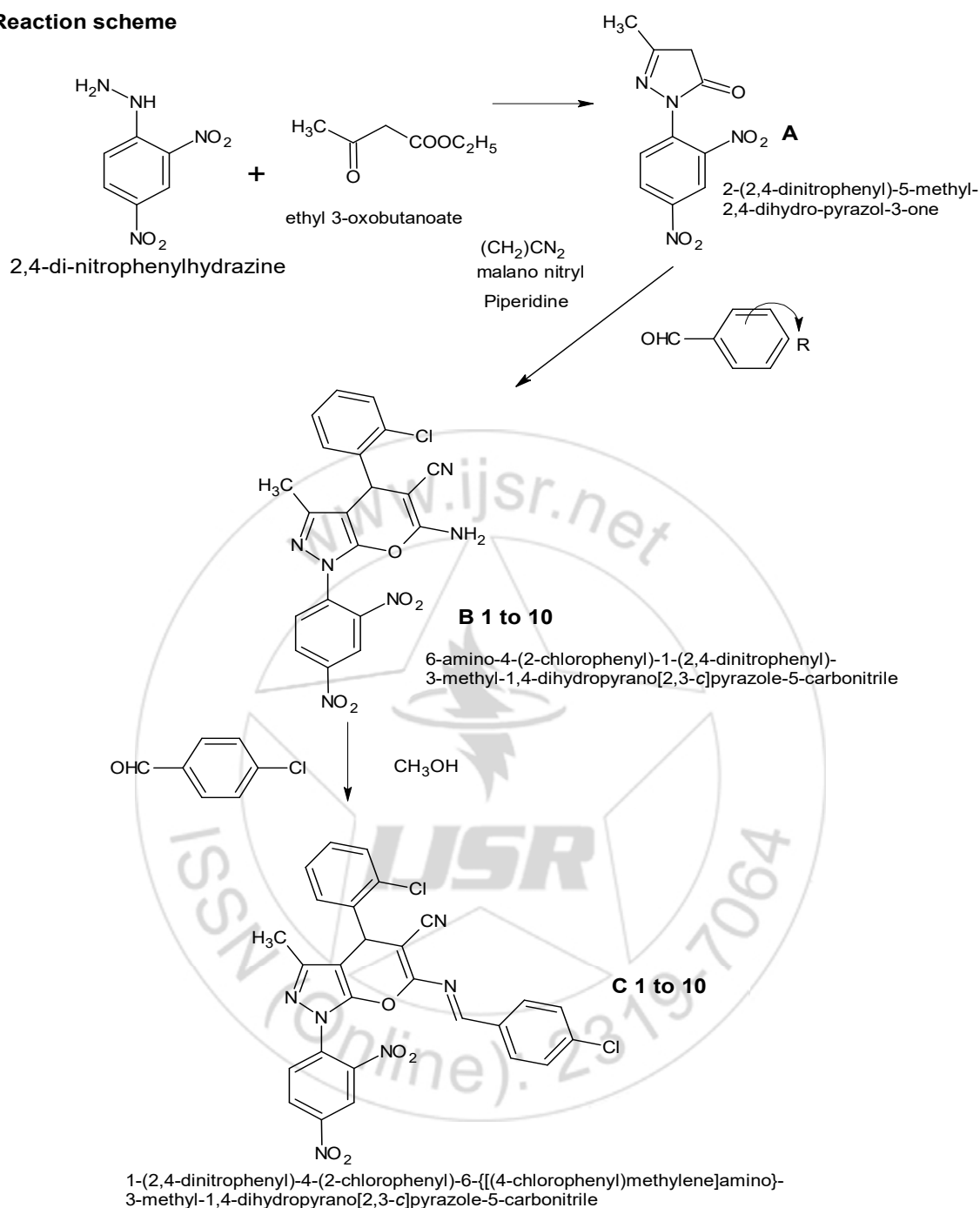
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recorded in DMSO on Bruker Advance II 400 MHz spectrometer using TMS as an internal standard. Purity of the compounds was checked by tlc on silica- G plates.

**Reaction scheme**

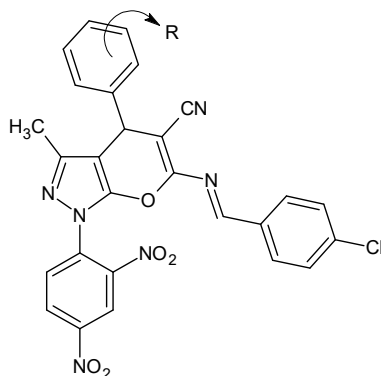


For compound A the elemental and spectral data was here they found element for  $C_{10}H_8N_4O_5$ : C (45.42%) H (3.03%) N (21.19%) and the calculated element for  $C_{10}H_8N_4O_5$ : C (45.46%) H (3.05%) N (21.21%) IR; (cm-1) :3079 (=CH), 2912 (-CH, Stretch), 1720 (>C=O), 1600 (>C=N Stretch), 1499 (>C=C<, aromatic ring), 1557 (-N=O), 1463 (-CH3 bend), 1343 (-C-N<), 1245 (>N-N<) .1H NMR (DMSO) :: 2.55, singlate (3H) (-CH3), 2.30, singlate (2H) (-CH2-), 8.16- 9.10, multiplate (3H) (Ar-H) .

IR; B-5 (cm) :3363 (>N-H), 3300 (-OH), 3050 (=C-H), 2941 (-C-H Stretch), 2198 (-C=N), 1592 (>C=N- Stretch),

1490 (>C=C< aromatic), 1543 (-N=O), 1442 (-CH 1H NMR (DMSO) ; SP-22 : 2.0346, singlate (2H) (-NH6.5825-8.3976, multiplate (7H) (Ar-H) . 3, bend), 1364 (C-N), 1270 (N-N), 1231 (-C-O), 1157 (C-O-C) . 2), 2.5513, singlate (3H) (-CH3), 4.7233, singlate (1H) (>CH-), 6.5825-8.3976, multiplate (7H) (Ar-H) .

**Physical constant of 1- (2, 4-dinitrophenyl) -4-(substitutedphenyl) -6-[[4-(4-chlorophenyl) methylene] amino]-3-methyl-1, 4-dihydropyrano [2, 3-c] pyrazole-5-carbonitrile (C-1 to 10)**



sub	R	Formula	Mol. Wt g/mol	Yield %	MP °C	C%		H%		N%	
						found	req	found	req	found	req
C-1	4-Cl	C <sub>27</sub> H <sub>16</sub> Cl <sub>2</sub> N <sub>6</sub> O <sub>5</sub>	575.35	71	102	56.32	56.36	2.78	2.8	14.57	14.61
C-2	2-Cl	C <sub>27</sub> H <sub>16</sub> Cl <sub>2</sub> N <sub>6</sub> O <sub>5</sub>	575.35	73	138	56.3	56.36	2.75	2.8	14.56	14.61
C-3	3-OCH <sub>3</sub> , 4-OCH <sub>3</sub>	C <sub>29</sub> H <sub>21</sub> ClN <sub>6</sub> O <sub>7</sub>	600.96	77	121	57.88	57.96	3.47	3.52	13.92	13.98
C-4	H	C <sub>27</sub> H <sub>17</sub> ClN <sub>6</sub> O <sub>5</sub>	540.91	70	110	59.9	59.95	3.15	3.17	15.48	15.54
C-5	2-OH	C <sub>27</sub> H <sub>17</sub> ClN <sub>6</sub> O <sub>6</sub>	556.91	78	152	58.15	58.23	3.03	3.08	14.02	15.09
C-6	3-OCH <sub>3</sub> , 4-OH	C <sub>28</sub> H <sub>19</sub> ClN <sub>6</sub> O <sub>7</sub>	586.93	73	98	57.24	57.3	3.21	3.26	14.27	14.32
C-7	4-OH	C <sub>27</sub> H <sub>17</sub> ClN <sub>6</sub> O <sub>6</sub>	556.91	75	161	58.16	58.23	3.05	3.08	15.01	15.09
C-8	4-N (CH <sub>3</sub> ) <sub>2</sub>	C <sub>29</sub> H <sub>22</sub> ClN <sub>6</sub> O <sub>5</sub>	583.98	70	78	59.98	59.64	3.75	3.8	16.73	16.79
C-9	4-OCH <sub>3</sub>	C <sub>28</sub> H <sub>19</sub> ClN <sub>6</sub> O <sub>6</sub>	570.94	80	85	58.94	58.9	3.3	3.35	14.65	14.72
C-10	3-NO <sub>2</sub>	C <sub>27</sub> H <sub>16</sub> ClN <sub>6</sub> O <sub>7</sub>	585.91	76	131	55.28	55.35	2.71	2.75	16.66	16.73

### 3. Anti Bacterial Activity

Synthesized compounds suggests their moderate antibacterial and antifungal activity as compared to the standard drugs Penicillin, Chloramphenicol, Streptomycin, Tetracyclin and Amphotericin using cupboar method. Antibiotic solution is prepared in sterile distilled water. Penicillin: 12 units, Chloramphenicol: 30 µg/ml, Streptomycin: 30 µg/ml, Tetracycline: 30 µg/ml, Fluconazole: 30 µg/ml

### Culture Activation

The culture was activated in nutrient broth and potato dextrose broth for bacteria and yeast respectively. One colony of each organism was inoculated and incubated at 37<sup>0</sup>C (bacteria) and 28<sup>0</sup>C (yeast) temperature for 24 hours. 200 µl of activated culture was inoculated in 25 ml of molted Nutrient agar and Yeast peptone agar for bacteria and yeast respectively. After proper mixing of culture it was poured in sterile 100mm Petri dish.

Synthesized compounds suggest their moderate antibacterial and antifungal activity as compared to the standard drugs Penicillin, Chloramphenicol, Streptomycin, Tetracyclin and Amphotericin.

**Table 2:** Antimicrobial activity of 1- (2, 4-dinitrophenyl) -4- (substitutedphenyl) -6- { [(4-chloroophenyl) methylene] amino} -3-methyl-1, 4-dihydropyrano [2, 3-c] pyrazole-5-carbonitrile

Sample code	Microorganisms							Yeast
	E.coli NCIM 2066	S.aureus MTCC 737	B.spizizenii MTCC 441	P.aeruginosa MTCC 1688	S.paratyphi A MTCC 735	B.pumillus MTCC 1607	K.pneumoniae MTCC 432	C.albicans MTCC 227
C-1	19	18	20	14	16	18	18	20
C-2	17	22	21	12	17	20	17	20
C-3	18	17	17	NI	16	16	14	19
C-4	14	15	15	10	14	15	16	16
C-5	16	NI	16	11	18	16	18	17
C-6	18	18	14	17	19	17	17	21
C-7	17	18	16	14	20	17	16	17
C-8	17	20	15	12	20	16	20	18
C-9	18	17	17	15	17	15	19	19
C-10	16	16	22	15	16	21	17	20

Where, SD1=Penicillin, SD2=Chloramphenicol, SD3=Streptomycin, SD4=Tetracyclin SD5= Fluconazole

#### 4. Conclusion

Over all evaluation of the synthesized compounds suggests their moderate antibacterial and antifungal activity as compared to the standard drugs Penicillin, Chloramphenicol, Streptomycin, Tetracyclin and Amphotericin.

Over all analysis of the results suggest that compounds C-1 was showed equal anti-bacterial activity for E.coli bacteria than the standard tested drugs streptomycine and other compounds have more activity then Chloreampenicol against E.coli NCIM 2066.

A compounds C-4 & 5 havea good anti-bacterial activity then standard tested drugs Chloramphenicol and streptomycine against S.aureus MTCC 737. A compound C-7, 9 and 10 compound showed very good anti-bacterial activity for P.aeruginosa than the standard tested drugs used for bio-assay and C-2 and C-8 have equal anti-bacterial activity then the standard tested drugs used for bio-assay against P.aeruginosa MTCC 1688. Compounds C-7, C-8 were showed high anti-bacterial activity then the standard tested drugs used for bio-assay for S.paratyphi A. And C-6 was show equal anti-bacterial activity then the standard tested drugs against S.paratyphi A MTCC 735. Compounds C-1, 2, 6, 7 and C-10 have High anti-bacterial activity then standard drugs Penicillin, streptomycine and Chloramphenicol for B.pumilus MTCC 1607. A compound SP-48 was showed High anti-bacterial activity for K.pneumoniae than the standard tested drugs used for bio-assay. And C-9 was showed equal anti-bacterial activity for K.pneumoniae than the standard tested drug Chloramphenicol against K.pneumoniae MTCC 432. A compound C-6 was showed equal anti-fungal activity then the standard tested drugs used for bio-assay for C.albicans. Remaining compounds against C.albicans MTCC 227.

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#### References

- [1] Schiff H., (1865), Ann. Chem., 3, 348.
- [2] Schiff H, (1864), Ann. Chem., 131, 118.
- [3] Tiwari S. S., Husain M. I. and Srivastava G. C., J. Ind. Chem. Soc., LVIII, 214 (1981) .
- [4] Dash B., Patra M. and Mahapatra P., J. Ind. Chem. Soc., LX, 772 (1983).
- [5] Profft Elmar and Hogel Egon., C. A. 80, 26913b (1974) .
- [6] Turan Zitouni G., Blache Y. and Guven K.; Bull. Chim. Farm, 140, 397 (2001).
- [7] Terzioglu N. and Gursoy A.; Eur. J. Med. Chem., 38, 781 (2003).
- [8] Trivedi D. K. and Desai K.R., (2005), Synthesis and biological evaluation of the 1- [4'-imino (substituted phenyl) phenyl] -2-phenyl-4- (4"-N:N-dimethylamino benzylidene) -imidazol-5-one., Oriental J. Chem., 21

(2), 345.

- [9] Pawar R. P., Andurkar N. M., J. Ind. Chem. Soc., 76 (5), 271 (1999) C.A., 137, 2711829y (1999)