



Research Article

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## Synthesis, Characterization and Antimicrobial activity of 6-amino-4-(substitutedphenyl)-1-(2,4-dinitrophenyl)-3-methyl-1,4-dihydropyrano[2,3-c]pyrazole-5-carbonitrile derivatives

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### ABSTRACT

6-amino-4-(substitutedphenyl)-1-(2,4-dinitrophenyl)-3-methyl-1,4-dihydropyrano[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-(2,4-dinitrophenyl)-5-methyl-2,4-dihydro-pyrazol-3-one 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 Cup borer method.

**Key words:** pyrazol-3-one, aldehyde, Ethenol, malononitrile. Piperidine.

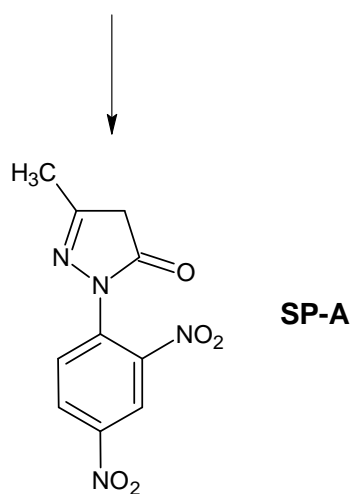
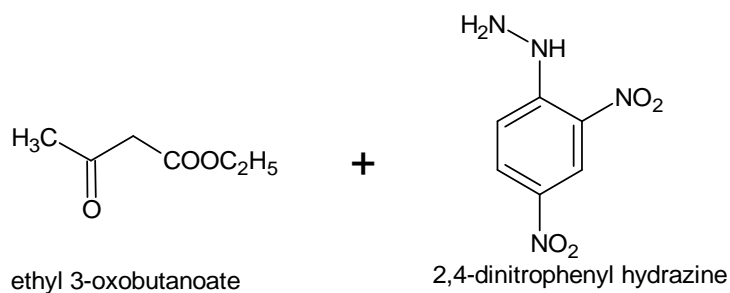
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### INTRODUCTION

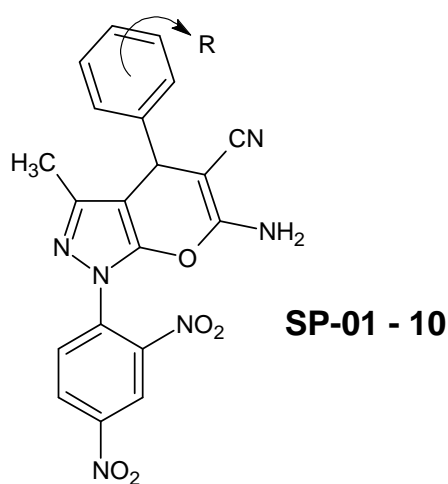
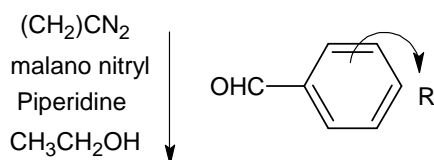
Pyrans are a six membered ring system having two double bond in conjugation and oxygen as hetero atom. Condensed pyrano[2,3-c]pyrazoles are biologically interesting compounds and their chemistry has received considerable attention[1-2]. Several pyrano[2,3-c]pyrazoles are reported to have useful biological effects such as analgesic and anti-inflammatory activities[3]. For the antimicrobial, insecticidal and anti-inflammatory activity of pyranopyrazole derivatives, See : El-Tamany et al. (1999) [4]; Ismail et al.(2003) [5]; Zaki et al. (2006) [6] Literature survey reveals that various pyran have proved to be potential drugs and a known to possess broad spectrum activities such as: antifungal[7-9], antiallergic[10], cytotoxic[11], antitumor[12], CNS active agents[13], antipyretic[14], anti invasive[15].

### EXPERIMENTAL SECTION

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

**Reaction Scheme**

2-(2,4-dinitrophenyl)-5-methyl-2,4-dihydro-pyrazol-3-one



6-amino-4-(substitutedphenyl)-1-(2,4-dinitrophenyl)-3-methyl-1,4-dihydropyrano[2,3-c]pyrazole-5-carbonitrile

**Synthesis of 2,4-di-Nitro Phenyl Pyrazolone (SP-A)**

For synthesis of SP-A, Mix together (0.4M) of redistilled Ethyl acetoacetate and (0.44M) of 2,4-di-Nitro Phenyl Hydrazine in a large evaporating dish. Heat the Mixture on boiling water bath in the fume cupboard for about 2 hrs and stir from time to time with a glass rod. Allow the heavy reddish syrup to cool the somewhat, add about 100 ml of ether and stir the mixture vigorously. The syrup which is insoluble in ether, will solidify within 15 minutes. Filter the solid at the pump and wash it thoroughly with ether to remove the coloured impurities. Recrystallise it from hot water or from a mixture of equal Volume of ethanol and water. The yield of the product was 76% and the product melts at 95°C. Found: C(45.42%) H(3.03%) N(21.19%), Calcd. for C<sub>10</sub>H<sub>8</sub>N<sub>4</sub>O<sub>5</sub>: C(45.46%) H(3.05%) N(21.21%) IR; SP-A (cm<sup>-1</sup>): 3079(=CH), 2912(-CH, Stretch), 1720(>C=O), 1600(>C=N Stretch), 1499(>C=C<, aromatic ring), 1557(-N=O), 1463(-CH<sub>3</sub> bend), 1343(-C-N<), 1245(>N-N<). <sup>1</sup>H NMR (DMSO); SP-A: 2.55, singlet (3H) (-CH<sub>3</sub>), 2.30, singlet (2H)(-CH<sub>2</sub>-), 8.16- 9.10, multiplet (3H) (Ar-H).

TABLE. NO. 1 Physical constant of 6-amino-4-(substitutedphenyl)-1-(2,4-dinitrophenyl)-3-methyl-1,4-dihydropyrano[2,3-c]pyrazole-5-carbonitrile

No.	Sub. No.	R	Molecular Formula	Mol. Wt. (g/m)	Yield (%)	M. P. °C	Carbon (%)		Hydrogen (%)		Nitrogen (%)	
							Found	required	Found	required	Found	required
1	SP-1	-4-Cl	C <sub>20</sub> H <sub>13</sub> ClN <sub>6</sub> O <sub>5</sub>	452.80	73	176	53.00	53.05	2.83	2.89	18.53	18.56
2	SP-2	-2-Cl	C <sub>20</sub> H <sub>13</sub> ClN <sub>6</sub> O <sub>5</sub>	452.80	76	136	53.00	53.05	2.83	2.89	18.53	18.56
3	SP-3	-3-OCH <sub>3</sub> , -4-OCH <sub>3</sub>	C <sub>22</sub> H <sub>18</sub> N <sub>6</sub> O <sub>7</sub>	478.41	71	156	53.17	55.23	3.74	3.79	17.52	17.57
4	SP-4	-H	C <sub>20</sub> H <sub>14</sub> N <sub>6</sub> O <sub>5</sub>	418.36	75	178	57.38	57.42	3.34	3.37	20.03	20.09
5	SP-5	-2-OH	C <sub>20</sub> H <sub>14</sub> N <sub>6</sub> O <sub>6</sub>	434.36	70	195	55.25	55.30	3.21	3.25	19.29	19.35
6	SP-6	-3-OCH <sub>3</sub> , -4-OH	C <sub>21</sub> H <sub>16</sub> N <sub>6</sub> O <sub>7</sub>	464.38	71	146	54.25	54.31	3.44	3.47	18.04	18.10
7	SP-7	-4-OH	C <sub>20</sub> H <sub>13</sub> N <sub>6</sub> O <sub>6</sub>	468.80	75	178	53.19	51.24	2.77	2.80	17.88	17.93
8	SP-8	-4-N(CH <sub>3</sub> ) <sub>2</sub>	C <sub>22</sub> H <sub>19</sub> N <sub>7</sub> O <sub>5</sub>	461.43	70	157	57.20	57.26	4.11	4.15	21.20	21.25
9	SP-9	-4-OCH <sub>3</sub>	C <sub>21</sub> H <sub>16</sub> N <sub>6</sub> O <sub>6</sub>	448.38	76	172	56.17	56.25	3.56	3.60	18.66	18.74
10	SP-10	-3-NO <sub>2</sub>	C <sub>20</sub> H <sub>13</sub> N <sub>7</sub> O <sub>7</sub>	463.35	71	182	51.77	51.84	2.79	2.83	21.10	21.16

TABLE. NO. 2 Antimicrobial activity of 6-amino-4-(substitutedphenyl)-1-(2,4-dinitrophenyl)-3-methyl-1,4-dihydropyrano[2,3-c]pyrazole-5-carbonitrile

Sr. No.	Sample code	Microorganisms							Yeast	
		<i>E. coli</i> NCIM 2066	<i>S. aureus</i> MTCC 737	<i>B. subtilis</i> MTCC 441	<i>P. aeruginosa</i> MTCC 1638	<i>S. paratyphi A</i> MTCC 735	<i>B. pumilus</i> MTCC 1607	<i>K. pneumoniae</i> MTCC 432	<i>C. albicans</i> MTCC 227	
0	SP-A	18	11	10	10	10	NI	12	13	
1	SP-01	15	19	14	NI	17	13	19	14	
2	SP-02	20	16	20	14	18	18	21	16	
3	SP-03	16	14	13	14	16	12	16	16	
4	SP-04	15	13	15	14	15	17	16	NI	
5	SP-05	18	15	12	10	14	11	14	15	
6	SP-06	19	20	14	NI	24	13	17	16	
7	SP-07	20	16	15	13	18	14	20	15	
8	SP-08	18	12	12	12	13	11	17	14	
9	SP-09	21	14	15	13	17	14	15	14	
10	SP-10	18	14	12	NI	18	14	16	15	

Note: The digits in above cell is indicates diameter for the zone of inhibition in milimeter (mm)

#### Preparation of 6-amino-4-(substitutedphenyl)-1-(2,4-dinitrophenyl)-3-methyl-1,4-dihydropyrano[2,3-c]pyrazole-5-carbonitrile (SP-01-10)

A mixture of 2-(2,4-dinitrophenyl)-5-methyl-2,4-dihydro-pyrazol-3-one (0.01M) malononitrile (0.01M) and substitutedbenzaldehyde (0.01M) in ethanol (40ml) and piperidine (1ml) was refluxed for 5 hours. After completion

of the reaction, the mixture was cooled and the resulting solid was crystallized from ethyl acetate. IR; SP-25 ( $\text{cm}^{-1}$ ): 3363 ( $>\text{N-H}$ ), 3300 ( $-\text{OH}$ ), 3050 ( $=\text{C-H}$ ), 2941 ( $-\text{C-H}$  Stretch), 2198 ( $-\text{C}\equiv\text{N}$ ), 1592 ( $>\text{C}=\text{N}$ - Stretch), 1490 ( $>\text{C}=\text{C}<$  aromatic), 1543 ( $-\text{N}=\text{O}$ ), 1442 ( $-\text{CH}_3$ , bend), 1364 ( $\text{C-N}$ ), 1270 ( $\text{N-N}$ ), 1231 ( $-\text{C-O}$ ), 1157 ( $\text{C-O-C}$ ).  $^1\text{H}$  NMR (DMSO); SP-22 : 2.0346, singlate (2H) ( $-\text{NH}_2$ ), 2.5513, singlate(3H) ( $-\text{CH}_3$ ), 4.7233, singlate(1H) ( $>\text{CH-}$ ), 6.5825-8.3976, multiplate(7H) ( $\text{Ar-H}$ ).

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