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Der Chemica Sinica, 2012, 3(4):830-833

Der Chemica Sinica
Pelagia Research Library
ISSN: 0976-8505
CODEN (USA) CSHIA5

Synthesis, spectral and microbial studies of 4-(substitutedphenyl)-1-(2,4-dinitrophenyl)-3-methyl-4,5-dihydro-pyrazolo[3,4-*d*]pyrimidin-6-ol

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ABSTRACT

4-(substitutedphenyl)-1-(2,4-dinitrophenyl)-3-methyl-4,5-dihydro-pyrazolo[3,4-d]pyrimidin-6-ol have been prepared by the refluxation for Seven hours of 2-(2,4-dinitrophenyl)-5-methyl-2,4-dihydro-pyrazol-3-one, substitutedbenzaldehyde and Urea in presence of hydrolichloric acid and methanol 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 ^{1}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, Pyrazolopyrimidinone, Methenol, Urea, DMSO.

INTRODUCTION

Pyrimidine is a six-membered heterocyclic compound consisting of two nitrogen atoms at 1 and 3 position of heterocyclic ring. Pyrimidine derivatives generally studied are 2-hydroxy pyrimidine, 2- mercapto pyrimidine and 2-amino pyrimidine. Pyrimidines have been isolated from the nucleic acid hydrolysates. Pyrazolopyrimidinone derivatives have attracted the attention of numerous researchers over many years due to their important biological activities [1–3]. The recent wide applications of pyrimidine derivatives as anti-tumor[4], anti-HIV-1 [5], analgesic [6], anti-depressive [7], anti-convulsant [8], anti-microbial [9], herbicides [10,11], anti-inflammatory and antioxidant [6,12] in progress.

MATERIALS AND METHODS

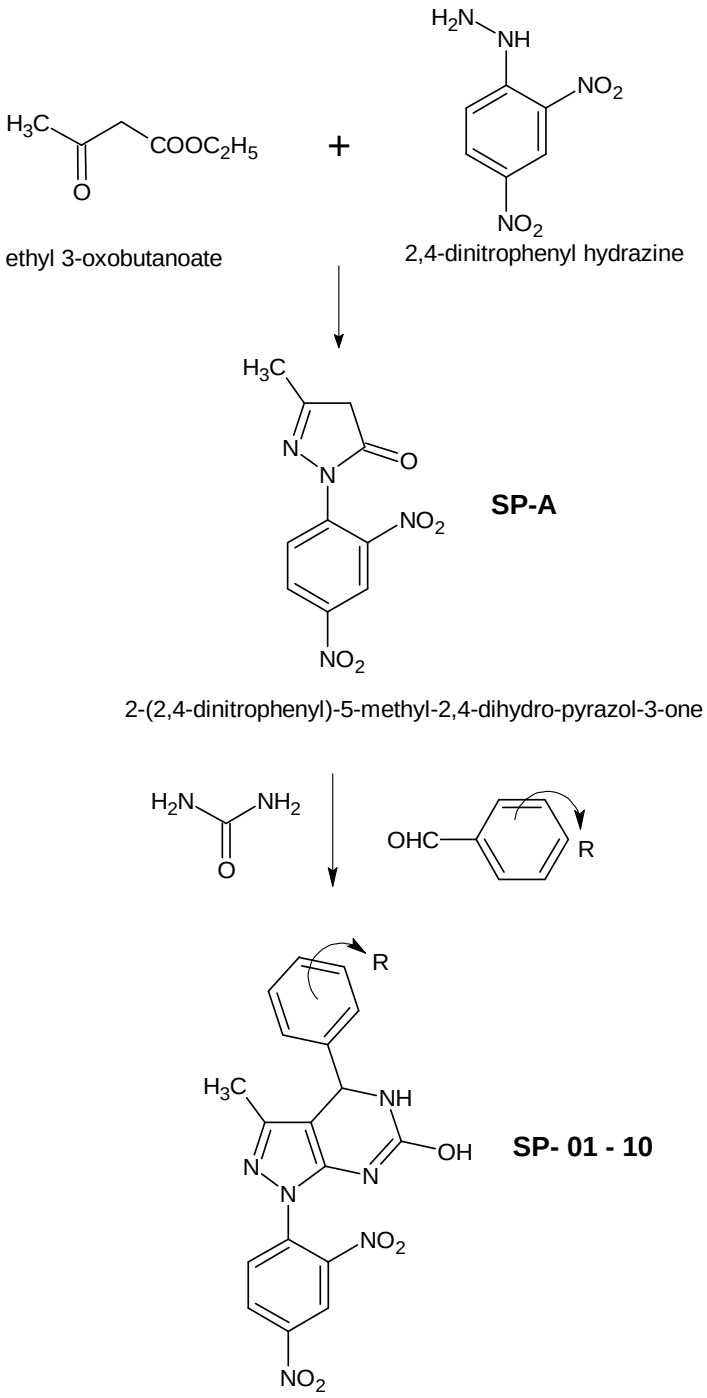
Melting points were taken in open capillary tube and were uncorrected. IR spectra were recorded on I.R. Spectrophotmeter of Bruker scientific Model No. Alpha E and instrument used for NMR Spectroscopy was recorded in DMSO on Bruker Advance II 400 MHz spectrometer using TMS as an internal standard. Purity of the compounds was checked by tlc on silica- G plates.

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 road. 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 95^0C. Found: C(45.42%) H(3.03%) N(21.19%), Calcd. for $C_{10}H_8N_4O_5$: C(45.46%) H(3.05%) N(21.21%) IR; SP-A (cm^{-1}): 3079(=CH), 2912(-CH, Stretch),1720(>C=O), 1600(>C=N Stretch), 1499(>C=C<, aromatic ring), 1557(-N=O), 1463(-CH_3 bend), 1343(-C-N<), 1245(>N-N<).^{1}H NMR (DMSO); SP-A: 2.55, singlate (3H) (-CH_3), 2.30, singlate (2H)(-CH_2-), 8.16- 9.10, multiplate (3H) (Ar-H).

Reaction Scheme



Preparation of 4-(substitutedphenyl)-1-(2,4-dinitrophenyl)-3-methyl-4,5-dihydro-pyrazolo[3,4-*d*]pyrimidin-6-ol (SP-01-10)

A mixture of 2-(2,4-dinitrophenyl)-5-methyl-2,4-dihydro-pyrazol-3-one (0.01M), Urea (0.01M) and substitutedbenzaldehyde (0.01M) and few drop hydrochloric acid in methanol was refluxed for 7 hours. After completion of the reaction, the mixture was cooled and the resulting solid was crystallized from ethyl acetate. IR; SP-98 (cm^{-1}): 3383 (>N-H), 3273 (–OH), 3078 (=C-H), 2901 (-C-H Stretch), 1605 (>C=N- Stretch), 1507 (>C=C< aromatic), 1537 (-N=O), 1413 (-CH_3 bend), 1324 (C-N), 1261 (N-N), 1211 (-C-O), 1157 (C-O-C). ^{1}H NMR (DMSO); SP-98 : 1.9080, singlate(1H)(-NH), 2.1202, singlate (1H)(Ar-OH), 2.5848, singlate(3H)(-CH_3), 3.0513 singlate(6H) (-$N(CH_3)_2$), 5.1997 singlate(1H) (>CH-), 6.7227 - 8.9677 multiplate(7H) (Ar-H)

TABLE. NO. 1Physical constant of 4-(substitutedphenyl)-1-(2,4-dinitrophenyl)-3-methyl-4,5-dihydro-pyrazolo[3,4-d]pyrimidin-6-ol

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	SP-1	-4-Cl	$C_{17}H_{11}ClN_4O_5$	428.78	82	238	50.40	50.42	3.03	3.06	19.56	19.60
2	SP-2	-2-Cl	$C_{17}H_{11}ClN_4O_5$	428.78	78	198	50.39	50.42	3.04	3.06	19.58	19.60
3	SP-3	-3-OCH_3,-4-OCH_3	$C_{20}H_{18}N_6O_7$	454.39	79	260	52.81	52.86	3.93	3.99	18.43	18.49
4	SP-4	-H	$C_{18}H_{14}N_6O_5$	394.34	83	191	54.75	54.82	3.53	3.58	21.26	21.31
5	SP-5	-2-OH	$C_{18}H_{14}N_6O_6$	410.34	76	168	52.61	52.69	3.39	3.44	20.42	20.48
6	SP-6	-3-OCH_3,-4-OH	$C_{19}H_{16}N_6O_7$	440.36	70	149	51.75	51.82	3.61	3.66	19.00	19.08
7	SP-7	-4-OH	$C_{18}H_{14}N_6O_6$	410.34	84	218	52.61	52.69	3.40	3.44	20.40	20.48
8	SP-8	-4-$N(CH_3)_2$	$C_{20}H_{19}N_7O_6$	437.40	78	210	54.84	54.92	4.31	4.38	22.37	22.42
9	SP-9	-4-OCH_3	$C_{19}H_{16}N_6O_6$	424.36	80	194	53.72	53.77	3.75	3.80	19.74	19.80
10	SP-10	-3-NO_2	$C_{18}H_{13}N_7O_7$	439.33	85	276	49.16	49.21	2.94	2.98	22.28	22.32

TABLE. NO. 2 Antimicrobial activity of 4-(substitutedphenyl)-1-(2,4-dinitrophenyl)-3-methyl-4,5-dihydro-pyrazolo[3,4-d]pyrimidin-6-ol

Sr. No.	Sample code	Microorganisms							*Yeast*
		E.coli NCIM 2066	*S.aureus* MTCC 737	*B.spizinzenii* MTCC 441	*P.aeruginosa* MTCC 1688	*S.paratyphi A* MTCC 735	*B.pumillus* MTCC 1607	*K.pneumoniae* MTCC 432	*C.albicans* MTCC 227
0	SP-A	18	11	10	10	10	NI	12	13
1	SP-01	17	18	14	13	16	13	20	15
2	SP-02	20	17	19	NI	18	16	20	16
3	SP-03	18	16	16	12	15	14	16	17
4	SP-04	16	13	17	15	15	17	16	NI
5	SP-05	18	16	12	12	14	14	14	17
6	SP-06	21	18	16	NI	21	14	17	19
7	SP-07	15	15	17	NI	11	11	16	14
8	SP-08	18	14	16	14	14	13	17	14
9	SP-09	20	14	16	14	17	17	13	15
10	SP-10	20	15	12	17	18	15	16	19

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

Acknowledgements

The authors are thankful to the Principal Dr. A. S. Rathod, Gujarat Arts & Science College, Ahmedabad for providing research facilities.The author also thankful to UGC. Ganeshkhind, pune, for Teacher Research Fellowship.

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