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CONVENTIONAL AND MICROWAVE-ASSISTED SYNTHESIS OF IODO SUBSTITUTED PYRAZOLINES DERIVATIVES AND SCREENING OF THEIR ANTIBACTERIAL AND ANTIFUNGAL ACTIVITIES

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ABSTRACT

2 Iodo - 6 (5-Furan - 2yl) 1- phenyl -4-5-dihydro-1-H pyrazol-3yl) phenol. A 2- Iodo - 6 (5 Furan - 2yl) 1 - Methyl - 4 - 5 dihydro - 1 H Pyrazol - 3 yl) Phenol.B 2- Iodo - 6 (5 Furan - 2yl) 1 - Ethyl - 4 - 5 dihydro - 1 H Pyrazol - 3 yl) Phenol.C 2- Iodo - 6 (5 Furan - 2yl) 1 - Benzyl - 4 - 5 dihydro - 1 H Pyrazol - 3 yl) Phenol. have been synthesized by the reaction of (3 Iodo - 2 hydroxyphenyl) -3 (Furan - 2yl) Prop-2 ene - 1 one with phenyl Hydrazine in ethanol. These newly synthesized compounds have been characterised on the basis of m.pt, elemental analysis and infra red spectral studies. These derivatives screened for their antibacterial activities on Escherichia coli, Staphylococcus aureus, Pseudomonas aeruginosa ,and antifungal activities on Aspergillus niger. All compounds exhibited poted inhibited activities.

Keywords; pyrazole microwave, antifungal antibacterial.

Introduction

Pyrazoline and their derivatives haye been extensively explored for their biological,anti-filarial and anti-bacterial activities Various compounds such as alkaloids, antibiotics, vitamins, haemoglobin, harmones and a large number of synthetic drugs and dyes contains Heterocyclic ring system. Heterocyclic compounds may be classified into aliphatic and aromatic compounds. Only those compounds with 5 to 6 member heterocyclic ring which are stable and exhibit aromatic properties are considered to be true heterocyclic Taking into consideration, strong luminescence, and inflammatory and a wide spectrum in biological media of substituted pyrazolines, the scheme of proposed study is aimed at exploration of these compounds on some physicochemical aspects of their new properties The application of microwave irradiation is used for

carrying out chemical transformations ,which are pollution free and eco-friendly These compounds of. pyrazolines will be assayed antibacterial to check antibacterial activity against gram positive and gram negative bacteria.

Materials and Methods

Iodo-substituted pyrazolines from - 1- (3 Iodo - 2 hydroxyphenyl) -3 (Furan - 2yl) Prop-2 ene - 1 one and Hydrazine / Phenyl Hydrazine in ethanol medium.

Synthesis of 1- (3-Iodo - 2 hydroxyphenyl) - 3 (Furan -2yl) Prop- 2- en-1one.1- (3-Iodo - 2 hydroxyphenyl) ethanone (0.01M) and furfural (0.01M) was added in ethanol solvent (10 ml). To this mixture NaOH (40%, 5ml) solution was added dropwise with constant stirring. The mixture was kept overnight. Then the mixture was poured over crushed ice & little HCl. The product was filtered and recrystallized from ethanol to obtain compound(1). **2- Iodo - 6 (5 Furan - 2yl) 4, 5 dihydro 1- H Pyrazol - 3 yl) Phenol.** 1- (3-Iodo - 2 hydroxyphenyl) - 3 (Furan - 2yl) Prop - 2 ene - 1ones & Hydrazine (0.01 M) was dissolved in ethanol solvent (10 ml) . To this mixture pulverised KOH sol^{n} (0.03 M) was added & put conventional and microwave irradiation for 10 min . The cooled reaction mixture was diluted with water & acidified with conc. HCl. The product was filtered & recrystallized

from ethanol to obtaine compound (2). **2- Iodo - 6 (5 Furan - 2yl) 1 - Phenyl - 4 - 5 dihydro - 1 H (Pyrazol - 3 yl) Phenol.** 1- (3-Iodo - 2 hydroxyphenyl) - 3 (Furan - 2yl) Prop - 2 ene - 1ones & Phenyl Hydrazine (0.01 M) was dissolved in ethanol solvent (10 ml) & the mixture was assisted for microwave irradiation. To this mixture pulverised KOH sol^{n} (0.03 M) was added and microwave irradiation for 10 min. The cooled reaction mixture was diluted with water & acidified with conc. HCl. The product was filtered & recrystallized from ethanol to obtain compound . Similarly compounds A, B, C, were synthsised.

Synthesis of 2- Iodo - 6 (5 Furan - 2yl) 1 - Phenyl - 4 - 5 dihydro - 1 H Pyrazol - 3 yl) Phenol.

A] Synthesis of 2- Iodo - 6 (5 Furan - 2yl) 1 - Methyl - 4 - 5 dihydro - 1 H Pyrazol - 3 yl) Phenol.

B] Synthesis of 2- Iodo - 6 (5 Furan - 2yl) 1 - Ethyl - 4 - 5 dihydro - 1 H Pyrazol - 3 yl) Phenol.

C] Synthesis of 2- Iodo - 6 (5 Furan - 2yl) 1 - Benzyl - 4 - 5 dihydro - 1 H Pyrazol - 3 yl) Phen

In conventional method the yield is lower as compared to microwave irradiation

Results & Discussion

All synthesized compounds have been established on the basis of chemical properties, element analysis and spectral analysis. Purity of compounds were checked by TLC on silica gel coated plates and iodine vapours were used for visualization. The melting points were reported in Tempo melting point apparatus as well as observed in Thiel's tube by using open capillary. IR spectra were recorded on PE-983 IR spectrophotometer. The carbon and hydrogen analysis was carried out on Carlo Erbo 1060 analyser. The nitrogen estimation was done on column N analyser 29.

Physical Characterization

Comp.	Molecular Formula	Molecular Weight	C %	H %	N %	I %	M.P. O^0C	Yield %
A	$C_{13}H_9IO_3$	340	45.88 (45.38)	2.64 (2.17)	-	37.35 (37.17)	121	76%
B	$C_{13}H_{11}IO_2N_2$	354	44.06 (44.54)	3.10 (3.25)	7.90 (8.12)	35.90 (35.47)	68	61.2%
C	$C_{19}H_{16}IN_2O_2$	431	52.90 (52.87)	3.71 (3.67)	6.49 (6.52)	67.24 (67.38)	120	81.6%

Compounds gave intense violet colouration with Ferric Chloride indicating presence of free Phenolic - OH group. It dissolved in dilute NaOH indicating presence of phenolic - OH group and gave deep blue colouration with concentrated H_2SO_4 showing absence of -CO-CH = CH – group .A paper

soaked with solution of in benzene turned bluish green when exposed to bromine vapours (knorr test for pyrazoline).

Infrared spectra of compounds

Compound A	Compound B	Compound C		Assignment
3395	3739	3392	——	OH
1697	——	——	——	C=C
3115	3126	3090	——	C-H
1163	1155	1155	——	C-O
1637	1530	1535	——	C=O
——	1605	1599	——	C = N
608	697	691	——	C-Cl

Infrared measurements for the compounds were made between 4000 to 650 cm-1 in KBr phase/Nujol mull with the help of spectrophotometer. Pertinent IR data for these complexes were recorded in Table 2. The spectra of 2- Iodo - 6 (5 Furan - 2yl) 1 - Phenyl - 4 - 5 dihydro - 1 H Pyrazol - 3 yl) Phenol. of isonitroso-p-methykacetophenone shows multiple medium broad absorption bands over a wide range (3392cm^{-1} and 3090 cm^{-1}). The presence of absorption features in this region points out to the presence of strong intramolecular hydrogen bonding involving oximino hydrogen atom and the bonding involving oximino hydrogen atom and the carbonyl oxygen atom of the ligand.

The IR spectra of the ligand shows characteristic absorption at 1535, 1599 and 691 cm^{-1} which may be assigned to vC=O, vC=N and vC-Cl modes respectively. In complexes these bands splitting giving two or more than two peaks. These split of bands or the presence of additional bands in C=O, C=N and C-Cl region point out to the probability of a structure having trans configuration of the compounds.

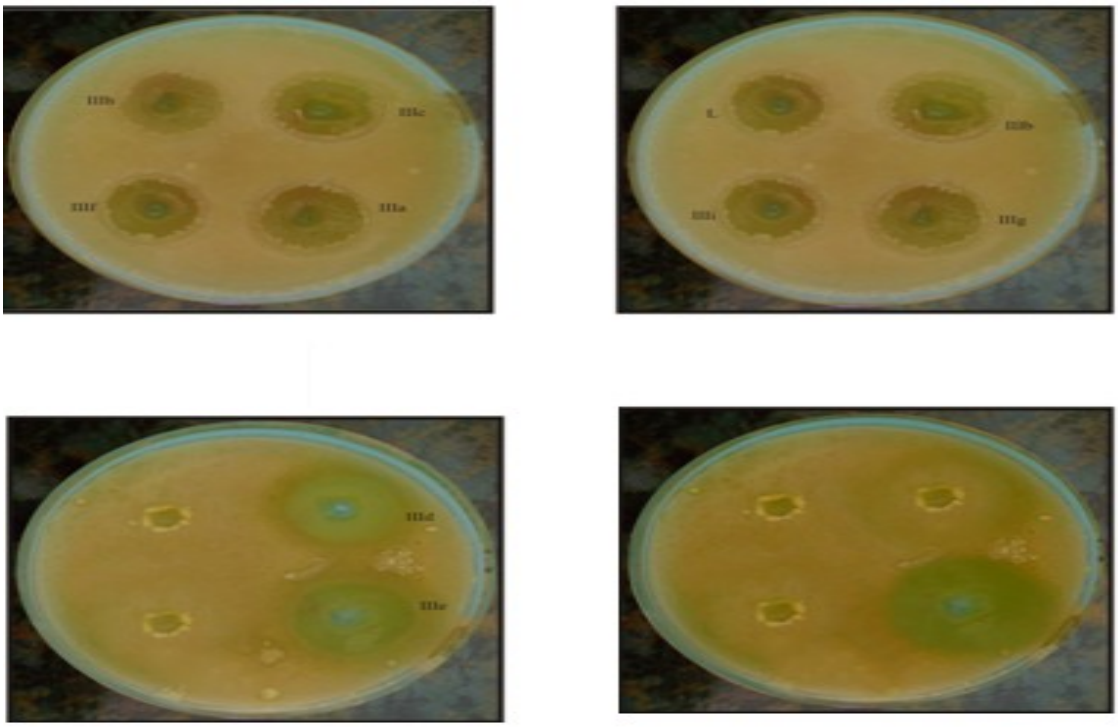
Antimicrobial Activity:-

Newly synthesized compounds were screened for their antibacterial activity against various strains of bacteria such as Bacillus megaterium, E.col, Pseudomonus aeruginosa ,Staphylocous aureus and anti fungal activity against Aspergillus niger, using cup plate agar diffusion method and muller Hinton agar as calture medium after 24 hrs of incubation at 37^0c. The zone of inhibition were measured in mm compound show high antibacterial activity.

The activity where compared with known reference drug like Amoxicillin Ciprofloxacin and Banzyl penicilline.

Zones of Inhibition in mm

Compound	**Antimicrobial activity**				**Antifungal Activity**
	B. Mega	**E.coli**	**P.aeruginosa**	**S.aereus**	**A.Niger**
	19	14	15	18	29
A	06	10	16	10	26
B	10	12	18	15	24
C	19	22	15	18	25
Amoxycillin	20	25	17	20	-
Benzylpenicilline	20	30	20	26	-
Ciprofloxacin	18	27	26	28	-
Ketoconazole	-	-	-	-	31



Conclusion:-

A novel series of SPD inhibitors were designed and synthesized and all compounds were characterized by molecular weight, IR spectra and TLC. All the data were found that the compound exhibited potent inhibitory activity against. In vitro which confirmed the mode of action of the target compound and provided direction for the design of molecules in the future.

The present study was carried out to develop a specific sensitive precise and accurate pharmaceutical tablet dose form as these substituted pyrazoline as it assayed antibacterial to check antibacterial activity against gram positive and gram negative bacteria it reveals that so much work has to be carried out on synthesis and complex formation of substituted pyrazoline with the help of pharmocology department.

References :-

Gupta R, Paul S, (1998) *Indian J Chem*, 37B:1211

Waldo J. P. , Mehta S. , (2008) *J. Org. Chem. Indian J Chem*, , 6660 - 6670.

Gupta Ragini Gupta Neetu , (2010) *Indian J Chem* Vol.49B, P.P. 351-355.

Koregaonkar, Patel S., (1996) *Indian J. Pharma. Sci.* 58 (6) , 222.

Rosa F. A. , Machado P., (2008) *Synlett*, 1673-1678.

C. Wu. Y. Fang, Lorack R.C. , F. Shi. (2010) *Org. Lett.*, 12, 2171 - 2173.

Borkhade K.T. and Marathe M.G., (1972) *Indian J. Chem.* 10, 48.

Ikhe S.A. and Narwade M.L., (2005) *Indian J Chem.* 44A, 1203.

Ikhe S.A., Rajput P.R. , (2005) *Indian J. Chem.* 44A, 2495.

Rekhas & Nagasundara K.R., (2006) *Indian J. Chem.* 45A, 2421-2425.

Agrawal N. N. and Soni P.A. (2005) Chemlnform online 22 Mar.

Nada M. Abunada, Hamdi M. Hassaneen, Nadia G. Kandile, Omar A. Miqdad (2008) *Molecules* 13, 1011-1024.