Synthesis & Analysis of Some Novel Amine Based Heterocyclic Compounds and its Derivatives Prepare by Different Catalyst Concentration and Time Duration

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Abstract- *now a days the time duration and resources are very costly and there for we check the activity of regularly used acid or base catalyst for the reaction of preparation of Schiff base, generally acetic acid & sodium hydrogen sulfate used as a catalyst in different amount various factor are affected to the reaction like amount of catalyst, time duration and temp of the reaction solvent are used as a media they are affected to reaction time and amount of yield. we are study and check the activity of acetic acid and sodium hydrogen pallet and check the TLC in different solvent used as mobile phase and change the value of Rf TLC plate*

Keywords- substituted N-atom containing heterocyclic compounds with free amine group, aldehydes, Acids, Bases and check the activity of catalyst with time

I. INTRODUCTION

The Schiff base-is make from the amine & adehyde the presence of Acid or base catalyst. in this type synthesis mostly the weak acid like acetic acid and weak base like sodium hydrogen sulfate type catalyst use ,basically we work on the check the activity of acid catalyst for making a novel heterocyclic Schiff base of pyridine & pyridines derivatives. the pyridine and pyridine ring heterocyclic compounds generally good biological activity on developing on anti malaria, anti viral, activity, Schiff bases containing pyridines derivative synthesized by different or modified method. The nitrogen contenting moiety generally good anti-oxidant property as well as good UV-boosters.

Experimental section:

Chemicals & analytical service :all starting material are purchased from industry they purity grade is low there for they are recrysttline from suitable solvents and check the solubility in different solvents, the TLC plate is made by Meark lab.usa for reaction monitoring, molecular structure determination by spectroscopy method, the check the TLC

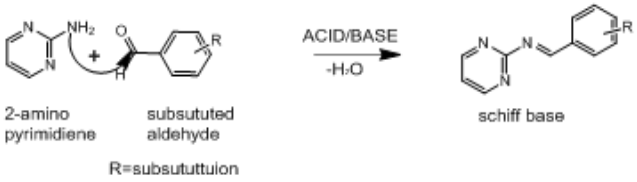
Materials & Solvents: 2-aminoyrimidine, 4-aminopyrimidine synthesis of chemicals: first take 25ml RBF and 24 and condenser they are 29 and std. joint on REMI magnetic stirrer set the minimum RPM all assembly fitted in oil bath set temp as per requirement starting martial are and aldehydes mix and then add minimum amount of suitable solvent adding and then add catalyst some amount and check activity of catalyst, then refluxed for required time and check the reaction monitoring on TLC plate different mobile phase ratio and set solvent ration for good Rf value

Analysis: the num was hydrogen present in compound by the 1HNMR made by 400HZ BRUCKER in DMSO as a solvent ,the13CNMR also determine by same instrument and same solvent ,The IR by FT-IR by SHIMAZU FT-IR. determine mass of compounds by mass spectroscopy .

Process for Acid -catalyst reaction: first take acetic acid and they are std. by NaOH & phenoplhelin as a indicator. the color change is colorless to pink .the check the % and mmole of acid catalyst for reaction. we check the % of conc. by tradition titration method.

process for compounds :take material 2-amino Pyrimidine(25 mmole) and benzalyde(25mmole) mix it then add solvent 4 ml solvent and add acetic acid as catalyst. stirring for different time and temp as per requirements and check the reaction on TLC plate.

1)TLC in Ethayl acetate & Hexane
2)TLC in toluene & Acetone



the above Reaction of the amine based heterocylic compounds and different R-CHO

Serial Num	code	Aldehydes	Melting point
1	2A	3-Nitro	180*C
2	2B	2,4 dihydro	200*C
3	2C	Benzaldehyde	160*C
4	2D	4-hydroxy	143*C
5	2E	2-nitro	168*C
6	2F	3-methoxy	115*C
7	2G	4-fluro	178*C
8	2H	3-bromo	122*C
9	2I	4-methoxy	129*C
10	2J	2,4 dicholro	152*C

Results & Discussion:Acid catalyst reaction & its rate of reaction change with time and temp conc. of catalyst Result chart is as below in this the three different chart are shown the above part of the paper they indicates they results are good at the highest temp and 20mmole the acid catalyst reaction .

Temp :Room temp , time :20 hrs ,catlyst: acetic acid,

CODE	10 mmole time	yield	15mm ole,time	yield	20 mmole time	Yield in %
2A	20	64	18	69	15	71
2B	20	67	18	76	15	80
2C	20	68	18	78	15	82
2D	20	63	18	72	15	84
2E	20	62	18	74	15	80
2F	20	64	18	76	15	81
2G	20	67	18	78	15	83
2H	20	68	18	74	15	84
2I	20	65	18	76	15	82
2J	20	64	18	70	15	79

Temp :80*C, time :15 hrs ,catlyst: acetic acid,

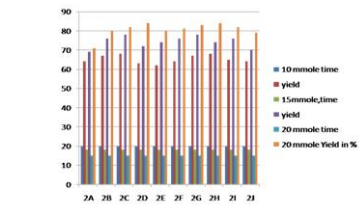
CODE	10 mmole time	yield	15m mole time	yield	20 mmole time	Yield in %
2A	15	70	12	75	9	92
2B	15	76	12	81	9	97
2C	15	72	12	77	9	98
2D	15	68	12	74	9	96
2E	15	76	12	76	9	97
2F	15	79	12	84	9	92
2G	15	70	12	76	9	96
2H	15	72	12	74	9	97
2I	15	74	12	78	9	95
2J	15	72	12	80	9	96

Temp :50*C, time :18 hrs ,catlyst:10 mmole acetic acid,

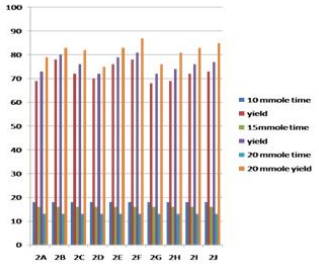
CODE	10 mmole time	yield	15m mole time	yield	20 mmole time	yield
2A	18	69	16	73	13	79
2B	18	78	16	80	13	83
2C	18	72	16	76	13	82
2D	18	70	16	72	13	75
2E	18	76	16	79	13	83
2F	18	78	16	81	13	87
2G	18	68	16	72	13	76
2H	18	69	16	74	13	81
2I	18	72	16	76	13	83
2J	18	73	16	77	13	85

ACID CATALYST

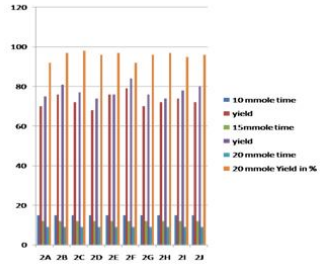
Temp :Room temp , time :20 hrs ,catlyst: acetic acid,



Temp :50*C, time :18 hrs ,catalyst:acetic acid,



Temp :80*C, time :15 hrs ,catalyst: acetic acid,



Temp :Room temp , time :20 hrs ,catlyst:NaHSO3

CODE	10 mmole time	yield	15mm ole,tim e	yield	20 mmole time	Yield in %
2A	21	52	18	65	15	69
2B	21	56	18	67	15	73
2C	21	54	18	61	15	64
2D	21	51	18	69	15	72
2E	21	54	18	58	15	75
2F	21	52	18	61	15	70
2G	21	57	18	67	15	73
2H	21	54	18	63	15	68
2I	21	53	18	59	15	62
2J	21	59	18	60	15	66

Temp :50*C, time :18 hrs , catalyst: $NaHSO_3$

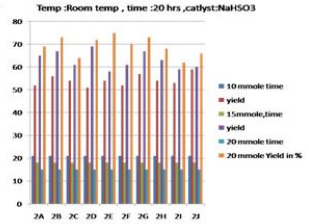
CODE	10 mmole time	yield	15mm ole time	yield	20 mmol e time	yield
2A	18	68	16	70	13	80
2B	18	75	16	79	13	85
2C	18	72	16	72	13	80
2D	18	64	16	69	13	85
2E	18	72	16	76	13	83
2F	18	67	16	73	13	81
2G	18	64	16	72	13	84
2H	18	62	16	67	13	86
2I	18	66	16	73	13	83
2J	18	69	16	79	13	87

BASE CATALYST

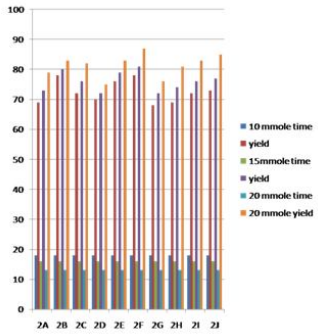
Temp :80*C, time :15 hrs ,catlyst:$NaHSO_3$

CODE	10 mmole time	yield	15mm ole time	yield	20 mmol e time	Yield
2A	15	71	12	73	9	91
2B	15	78	12	81	9	93
2C	15	73	12	79	9	94
2D	15	67	12	72	9	90
2E	15	75	12	79	9	92
2F	15	72	12	76	9	85
2G	15	66	12	74	9	84
2H	15	64	12	70	9	81
2I	15	68	12	77	9	82
2J	15	73	12	82	9	86

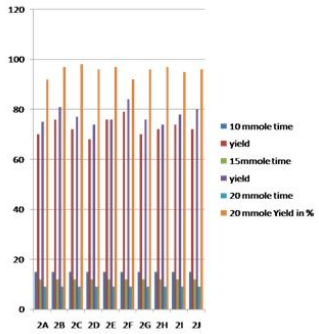
Temp :Room temp , time :20 hrs ,catlyst:NaHSO3



Temp :50*C, time :18 hrs ,catalyst:acetic acid,



Temp :80*C, time :15 hrs ,catalyst: acetic acid,



Analysis Report of Series 2A to 2J IR By FT-IR,1HNMR,13CNMR & MASS Fragmentation DATA)

(2A) By FT-IR: 1600-1500 cm-1 ,-C=C-Aromaticstretching3050cm-,1-C=N stretching1595cm1,1HNMR(400MHz,DMSO) δ=7.50, δ=7.98 to8.01, δ=7.37 to7.40, δ=7.77 to7.81, δ=8.80 to 8.85 δ=8.95,13CNMR(400MHz,DMSO), δ=60.76, δ=111.44, δ=121.12, δ=122.41, δ=129.79, δ=133.54, δ=158.12, δ=160.96, M/Z=[M+H]+=178.68

(2B)IR By FT-IR:-C-OH group 3550 to 3420cm-1,-C=C-Aromaticstretching3050cm-,1-C=Nstretching 1595cm-12140to2100cm-1(-C-NC,S),1HNMR(400MHz,DMSO) δ=6.72 , δ=7.32,δ=7.52,δ=8.85,δ=8.92,δ =9.32,13CNMR(400MHz,DMSO) δ=115.90, δ=117.58, δ=123.45 δ=131.25, δ=146.25, δ =149.50, δ =159.60,δ=160.17, δ=166.12, M/Z=[M+H]+=179.17

(2C)IR By FT-IR: -C=N stretching 1595cm-1 and -C=C-Aromatic stretching3050cm-1,H1NMR(400MHZ,DMSO) δ=7.02 to 7.26, δ=7.560 to 7.720, δ=8.16 to8.16, δ=8.46 to 8.49,13CNMR(400MHz,DMSO),δ=111.22,122.18,129.05,133 .05,154.38,160.01 M/Z=[M+H]+=148.1

(2D)IR By FT-IR:-C-OH group 3550 to 3220cm-1,-C=C-Aromaticstretching3050cm-,1-C=N-stretching 1595cm-12140to2100cm-1(-C-N,S),1HNMR(400MHz,DMSO) δ=6.56,6.62,δ=7.62,δ=8.82,δ=9.56,δ=9.62,13CNMR(400MHz ,DMSO)δ=113.90,δ=116.25, δ=129.30 δ=130.72, δ=158.60, δ=160.82, δ=167.12, M/Z=[M+H]+=163.18

(2E)IR By FT-IR: -C=N stretching 1595cm-1 and -C=C-Aromatic stretching3050cm-1 N=O1400to1600cm1,H1NMR(400MHz,DMSO)δ=7.30to7.65, δ=7.80(t,aero,C=6),δ=8.088.29,δ=8.72to8.85,δ=8.93,13CNM R(400MHz,DMSO),δ=110.14,δ=125.86,δ=131.25,δ=142.78δ =155.42,δ=160.20,δ=167.45,M/Z=[M+H]+=165.21

(2F)IR By FT-IR:CH3-O stretching 2810cm-1 ,-C=C-Aromatic stretching3050cm-,1-C=N stretching1595cm1,1HNMR(400MHz,DMSO) δ=3.80, δ=7.00to7.02, δ=7.37 to7.40, δ=7.77to7.81,δ=8.80to8.85δ=8.95,13CNMR(400MHz,DMSO),δ=60.76,δ=111.44, δ=121.12, δ=122.41, δ=129.79, δ=133.54, δ=158.12 δ=160.96,M/Z=[M+H]+=178.68

(2G)IR By FT-IR:-C-F,1400 to 1000 cm-1 ,-C=C- Aromatic stretching3050cm-,1-C=N stretching 1595cm-12140 to 2100 cm-1(-C-N-C -,S),1HNMR(400MHz,DMSO) δ=7.28 to 7.32, δ=7.56 , δ=7.96, δ=8.7, δ=9.42,13CNMR(400MHz,DMSO) δ=114.50, δ=116.25, δ=128.37, δ=130.24, δ=158.68, δ=164.25, δ=167.12, M/Z=[M+H]+=165.71

(2H)IR By FT-IR:-C-Br- 750cm-1 ,-C=C-Aromaticstretching3050cm-,1-C=Nstretching 1595cm-12140 to 2100 cm-1(-C-N-C -,S),1HNMR(400MHz,DMSO)δ=7.37 to7.40, δ=7.77to7.81,δ=8.67to8.85,δ=8.95,13CNMR(400MHz,DMSO)δ=113.22,δ=122.50,δ=127.70,δ=129.12,δ=132.67,δ=157.12, δ=163.16, M/Z=[M+H]+=226.12

(2I)IR By FT-IR:CH3-O stretching 2810cm-1 ,-C=C-Aromatic stretching3050cm-,1-C=N stretching 1595cm-12140 to 2100 cm-1(-C-N-C -,S),1HNMR(400MHz,DMSO) δ=3.80, δ=7.00to7.02, δ=7.37 to7.40, δ=7.77 to7.81, δ=8.85,δ=8.95,13CNMR(400MHz,DMSO), δ=60.76, δ=111.44, δ=121.12, δ=122.41, δ=129.79, δ=133.54, δ=158.12, δ=160.96, M/Z=[M+H]+=178.68

(2J)IR By FT-IR:-C-Cl,400 to 750 cm-1 ,-C=C- Aromatic stretching3050cm-,1-C=N stretching 1595cm-12140 to 2100 cm-1(-C-N-C -,S),1HNMR(400MHz,DMSO) δ=7.50 to 7.52,δ=7.70,δ=7.96,δ=8.80,13CNMR(400MHz,DMSO) δ=114.50, δ=127.70, δ=129.52 δ=131.30, δ=158.20, δ=160.32, δ=164.64, M/Z=[M+H]+=216.0

II. RESULTS AND DISCUSSION

the Schiff base make by the acid or base catalyst we check the activity of known catalysts are use for the reactions ,all Reaction rate is depend on the time and temperature they are very creative role in synthesis ,the acetic acid & sodiumsulfite are used as a catalyst at the as per previous regarding data of sample is 80℃ in most of paper, now a days the green synthesis path is growing research area in chemistry. there for our area of research is based on the minimum input of energy & catalyst .because of both are directly connected to the environment and cost related so; our work is the first we used acetic acid as a catalyst in the reaction the different mini mole concentration, time & temperature, they are gave the good efficiency of catalyst at regular as per previous paper study temp but they gave the 20mmole conc. of acetic acid gave the good yields, we show and review above tables and graph they clear indicate the acid catalyst gave good yield for the preparation of novel schiff base.

III. ACKNOWLEDGMENTS

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