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Microwave Assisted Solvent Free Synthesis of Substituted Aniloquinoline and its Antibacterial Study

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Abstract: ***Synthesis of linear dibenzonapthyridines is well known by two step conventional method through an intermediate aniloquinoline which was reported by K.J.R. Prasad et al. In the present work an attempt was made to synthesis dibenzonapthyridines through microwave method, in solvent free condition. We have also carried out antibacterial activity, which has given a good source of results. Simultaneously our effort to synthesis dibenzonapthyridines through one pot multicomponent was also attempted.***

Keywords: Aniloquinolines Microwaves In Organic Synthesis Antibacterial Activity

1. Introduction

- There are so many synthetic strategies are there for the synthesis of organic compounds.
  Example:
  Photochemical
  Electrochemical
  Sonochemical
  Microwave method
  Enzymatic method
- Microwave method is the best method, because easy availability of microwave sources.
- For microwave heating, the substance must possess a dipole moment.
- A dipole is sensitive to external electric field and tries to align itself with the field by rotation.
- If submitted to an alternating current, the electric field is inversed at each alterance and therefore dipoles tend to move together to follow the inversed electric field.
- The electric field of commonly used irradiation frequency **(2450 MHz)** oscillates **4.9×10^9** times per second.
- Thus, microwave heating is directly dependent on dielectric properties of a substance, dielectric constant (ε') and dielectric loss (ε''). The ability of a material to convert electromagnetic energy into heat energy at a given frequency and temperature, is calculated using

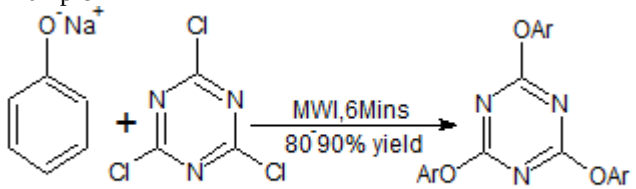
$$\varepsilon'' / \varepsilon' = \tan \delta$$

- Compounds with high dielectric constants, tend to heat rapidly under microwave irradiation, while less polar substances or compounds with no net dipole moment, as well as highly ordered crystalline substances, are poorly absorbing.
- Thus, polar molecules in a non-polar solvent would absorb energy, but not the solvent or the reaction vessel, the use of a solvent is not always mandatory for the transport of heat.
- Therefore, reactions performed under **solvent-free conditions** present an alternative in the microwave chemistry and constitute an environmentally benign technique, which avoids the generation of toxic residues, like organic solvents and mineral acids, and thus allows the attainment of high yields of products at reduced environmental costs.

Types of microwave assisted organic reactions:
The microwave-assisted organic reactions have been broadly classified into two categories:

a) Microwave-assisted reactions using solvents.
b) Microwave-assisted reactions using solvent-free conditions.

Microwave assisted reactions using solvent-free conditions
Example



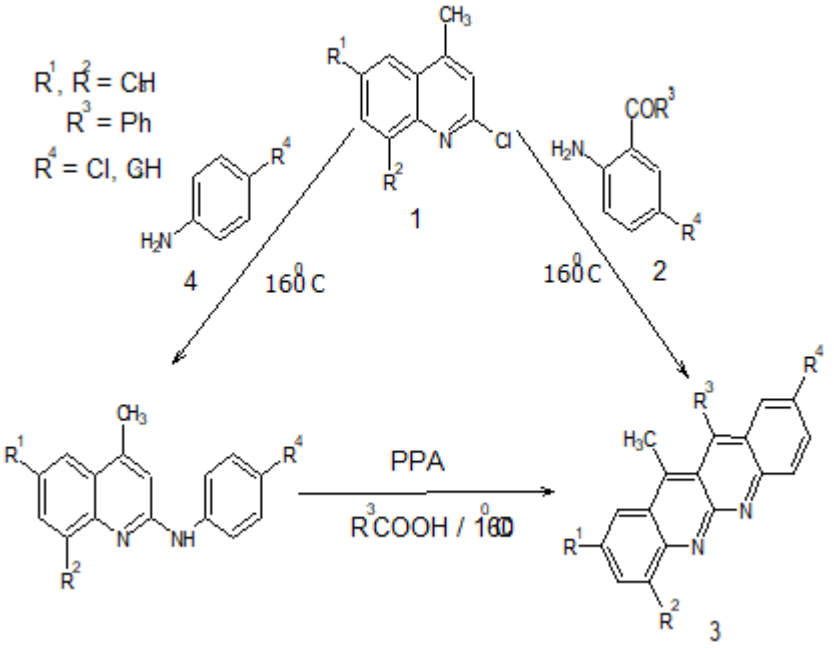
2,4,6-trichloro-1,3,5-triazine

2. Present Work

Scope of the present work

- Synthesis of linear dibenzonapthyridines is well known by two step conventional method through an intermediate aniloquinoline which was reported by K.J.R. Prasad et al.
- In the present work an attempt was made to synthesis dibenzonapthyridines through microwave method, in solvent free condition.
- We have also carried out antibacterial activity, which has given a good source of results. Simultaneously our effort to synthesis dibenzonapthyridines through one pot multicomponent was also attempted.

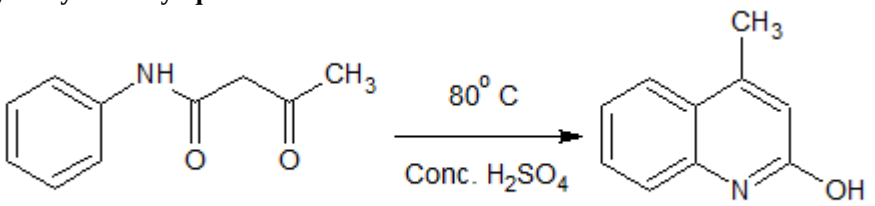
Scheme: Reaction sequence to achieve linear dibenzonapthyridines



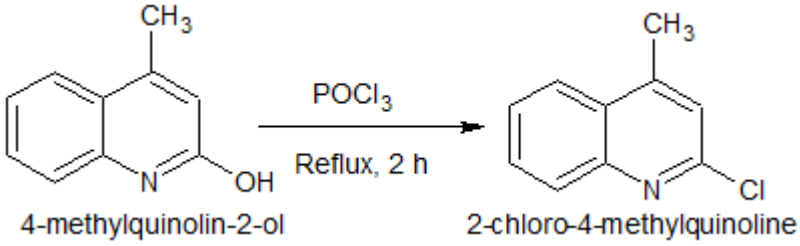
3. Experimental part

3.1 Conventional Method

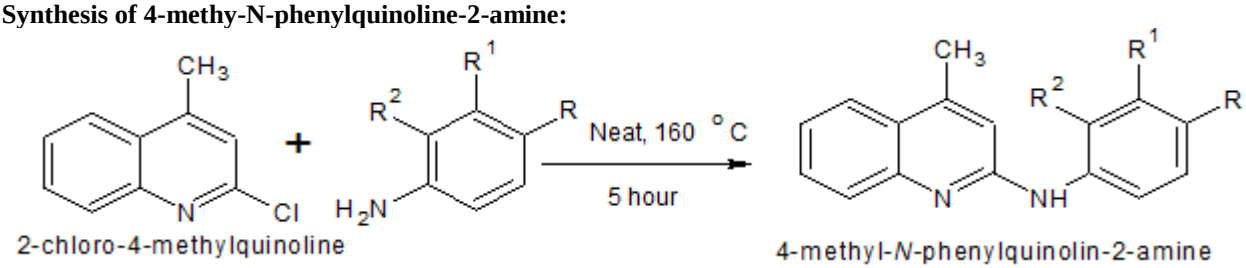
a) Synthesis of 2-hydroxy 4-methyl quinoline:



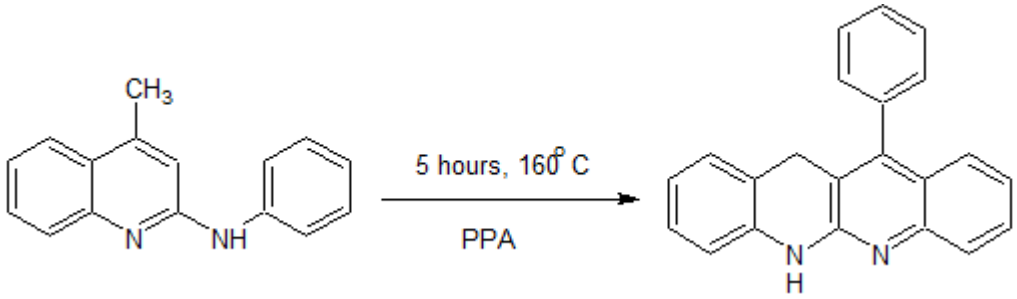
b) Synthesis of 2-chloro 4-methyl quinoline:



c) Synthesis of 4-methy-N-phenylquinoline-2-amine:

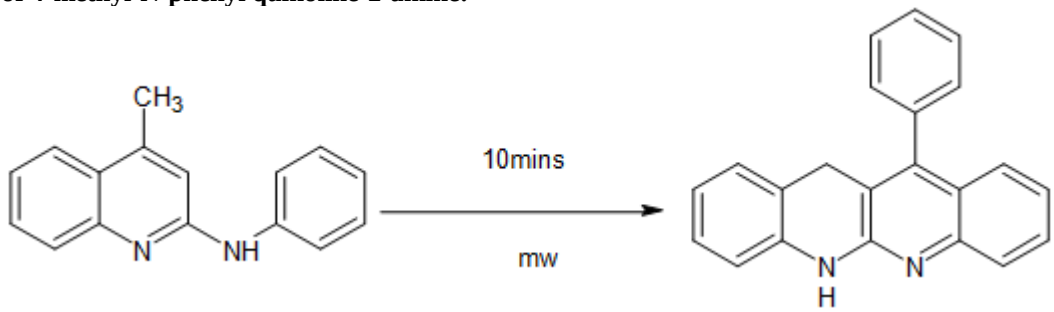


d) Synthesis of 10H-dibenzo[b,g]naphthol[1,2,3-de][1,8]naphthyridine:



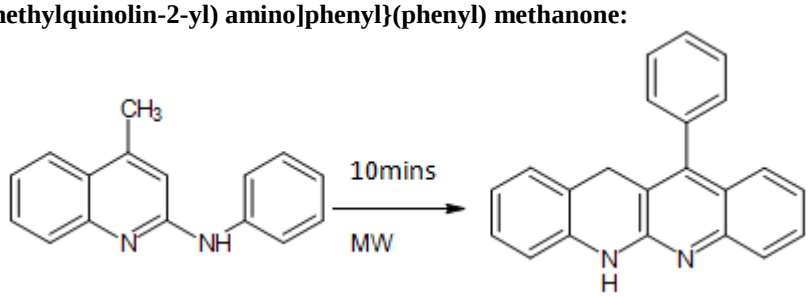
3.2 Microwave synthesis

a) Synthesis of 4-methyl-N-phenyl quinoline-2-amine:



	R	R^1	R^2
a	H	H	H
b	CH3	H	H
c	Cl	H	H
d	COOH	H	H
e	H	H	H
f	H	Cl	H

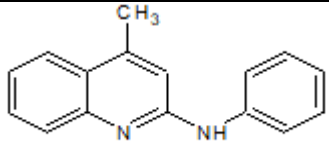
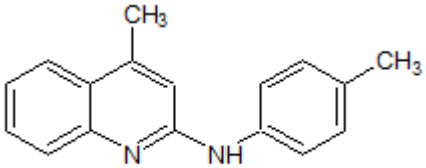
b) Synthesis of {2-[(4-methylquinolin-2-yl) amino]phenyl}(phenyl) methanone:



4. Results and Discussion

- An attempted synthesis of linear dibenzonapthyridine was unsuccessful under microwave method; the reaction resulted in the synthesis of unknown compound, a careful investigation of the resultant compound led to confirmation that the compound was one of the intermediate of the reaction.

Table 1: Conventional method

S. No.	Compound	Mol. formula	Mol.weight	Yield %
a	 4-methyl-*N*-phenylquinolin-2-amine	$C_{16}H_{14}N_2$	234.29	62%
b	 4-methyl-*N*-(4-methylphenyl)quinolin-2-amine	$C_{16}H_{16}N_2$	248.32	50%

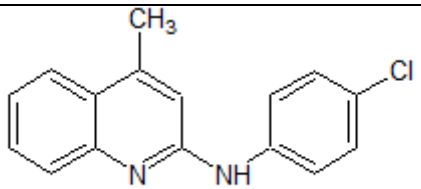
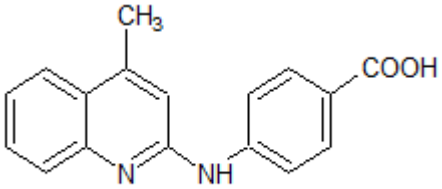
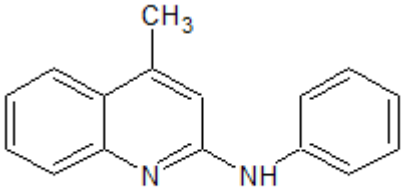
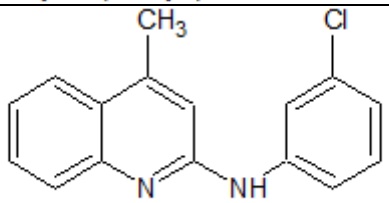
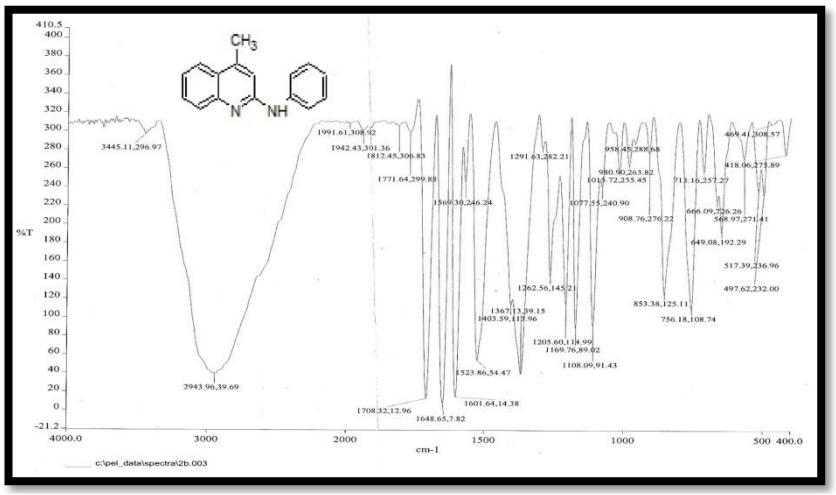
c	 *N*-(4-chlorophenyl)-4-methylquinolin-2-amine	$C_{16}H_{13}N_2Cl$	268.72	58%
d	 4-[(4-methylquinolin-2-yl)amino]benzoic acid	$C_{17}H_{14}N_2O_2$	278.3	61%

Table 2: Microwave method

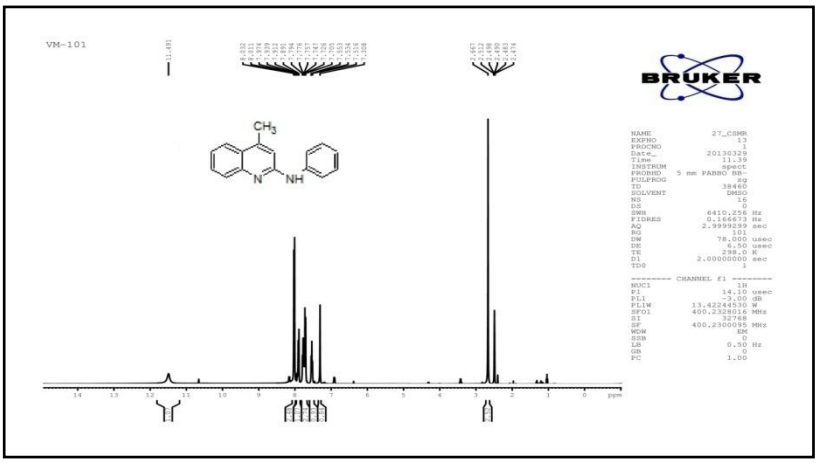
e	 4-methyl-*N*-phenylquinolin-2-amine	$C_{16}H_{16}N_2$	248.32	81%
f	 *N*-(3-chlorophenyl)-4-methylquinolin-2-amine	$C_{16}H_{13}N_2Cl$	268.72	85%

Spectral data of the synthesized compounds:
a) 4-methyl-N-phenylquinolin-2-amine:

IR spectra:



H^1 NMR spectra:



e) 4-[(4-methylquinolin-2-yl)amino]benzoic acid:

H^1 NMR spectra:

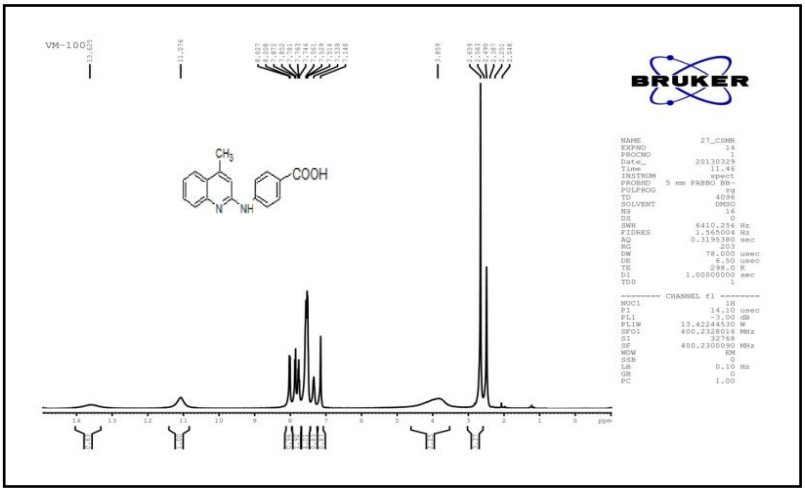
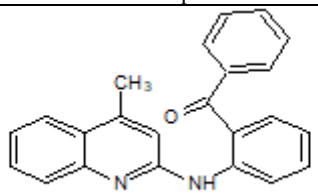
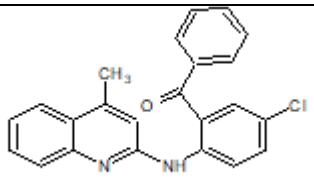
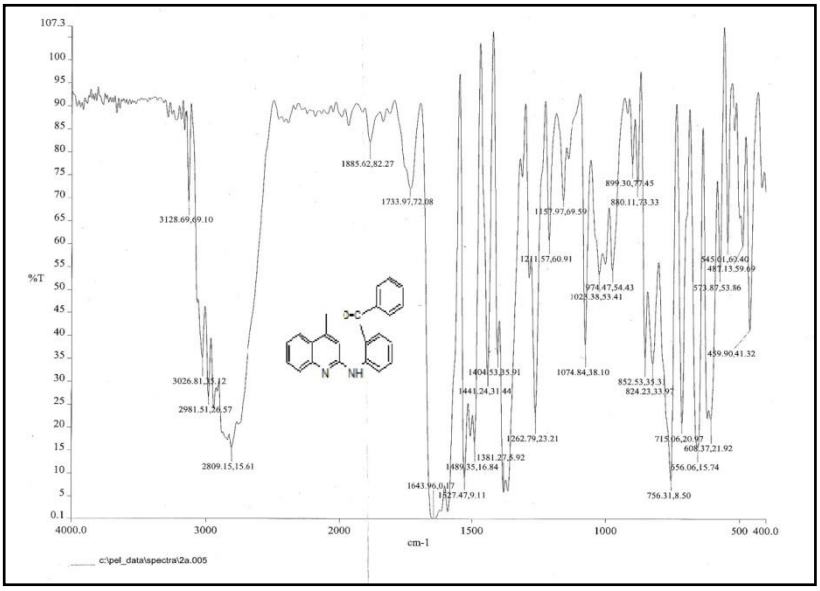


Table 3

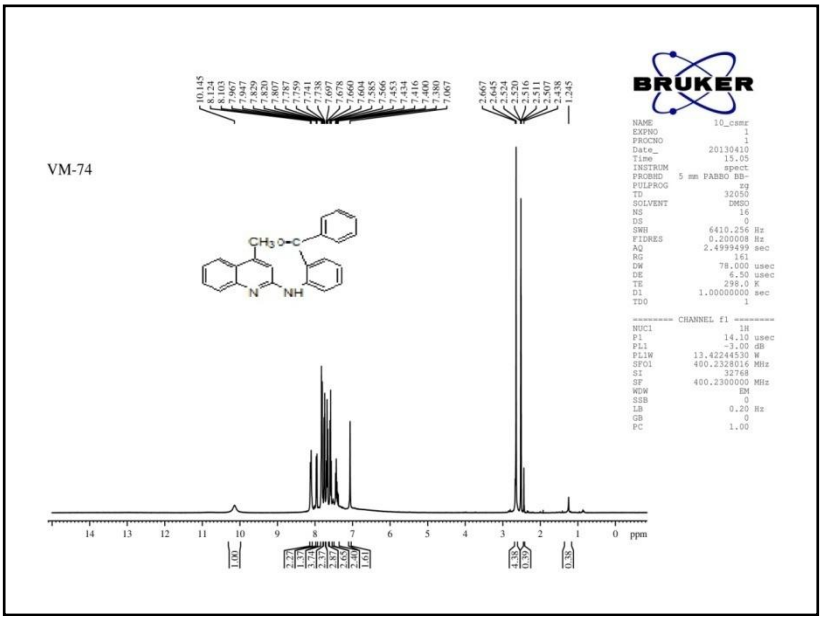
S.no.	Compound	mol. formula	Mol.weight	Yield %
g	 {2-[(4-methylquinolin-2-yl)amino]phenyl}(phenyl)methanone	$C_{23}H_{18}N_2O$	338.4	89%
h	 {5-chloro-2-[(4-methylquinolin-2-yl)amino]phenyl}(phenyl)methanone	$C_{23}H_{17}N_2ClN_2O$	372.8	82%

f) {2-[(4-methylquinolin-2-yl)amino]phenyl}(Phenyl) methanone:

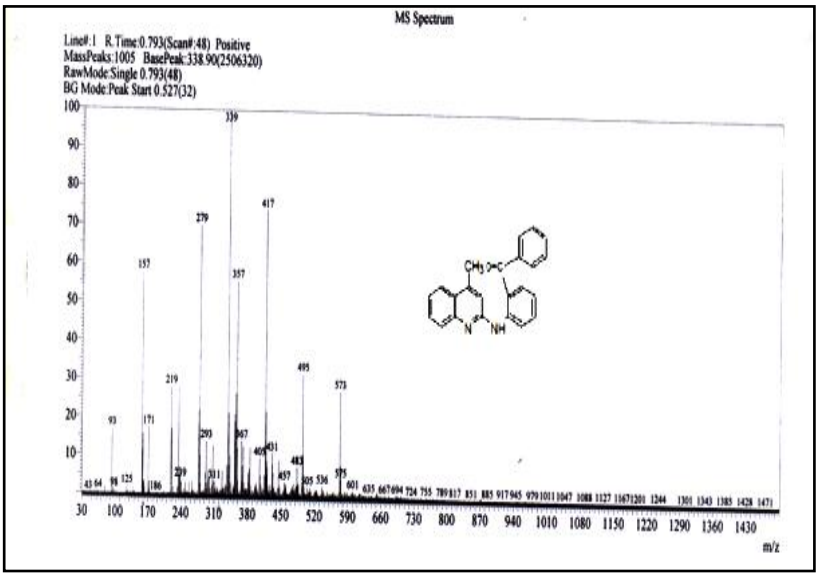
IR data



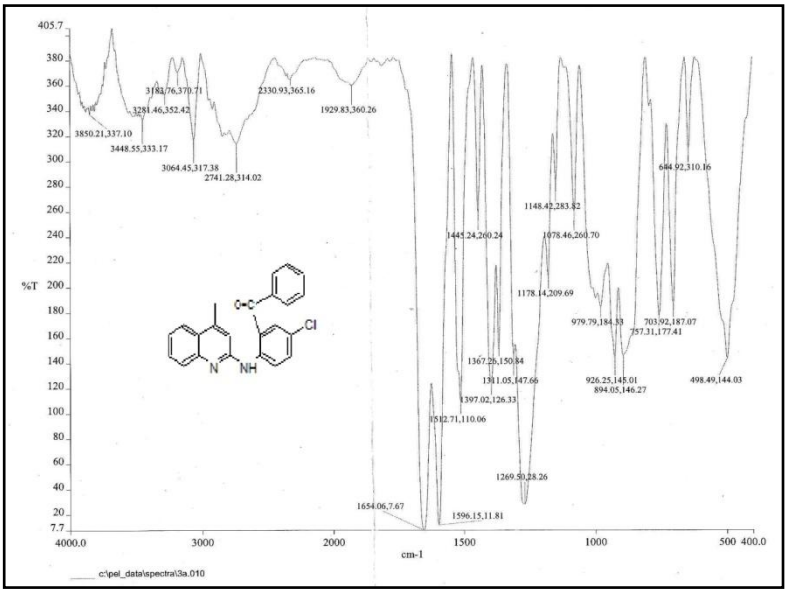
H[1] NMR spectra



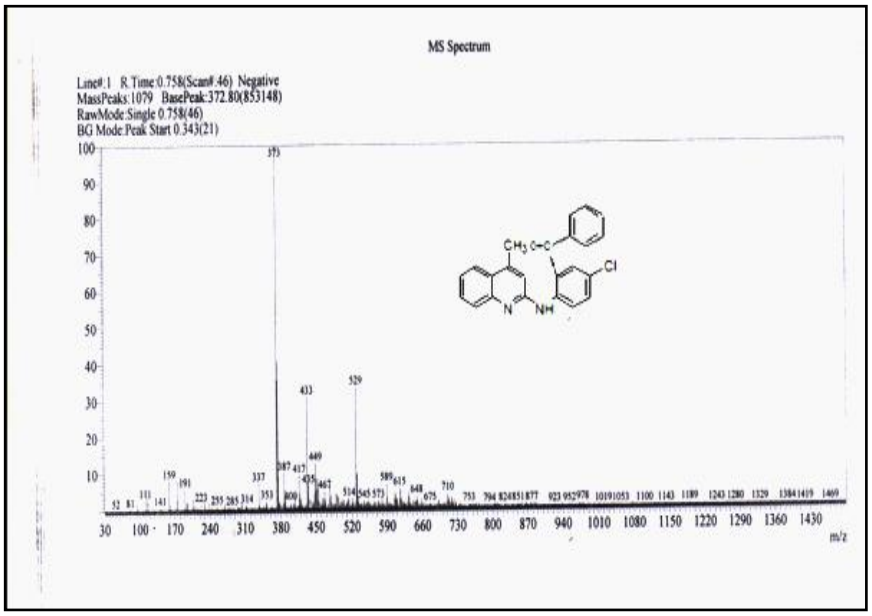
MASS spectra



g) {5-chloro-2-[(4-methylquinolin-2-yl)amino]phenyl}(phenyl)methanone: IR SPECTRA



MASS spectra



Antibacterial activity

5. Methodology

Gram positive bacteria namely staphylococcus aureus and bacillus subtilis and gram negative bacteria namely Eschrichia coli, pseudomonas aeruginosa and klebsiella pneumoniae were used as target bacteria.

The bacteria were screened for their sensitivity towards the plant extracts by Agar well diffusion method (Tepe et al., 2004). In this method, 24 hours old nutrient broth cultures of test bacteria were swabbed uniformly on solidified sterile nutrient agars using sterile cotton swab. Then, aseptically wells of 6 mm diameter were bored in the inoculated plates with the help of gel puncher and compound (1 mg/ml of DMSO), Standard (chloramphenicol, 1 mg/ml) and control (DMSO) were added into the respectively labeled wells. The plates were incubated at 37 ^{0}C for 24 hours in upright position and the zone of inhibition was recorded. The experiment was carried in triplicates to get average reading.

Table 1: Antibacterial activity

Treatment	Zone of inhibition in mm				
	E.Coli	P.aeruginosa	K.pneumoniae	B.subtilis	S.aureus
Control (DMSO)	-	-	-	-	-
Standard (chloramphenicol)	17	18	16	20	23
Compound (g)	9	11	10	12	15
Compound (h)	12	12	13	14	17

6. Conclusion

Finally we have described the efficient synthesis of aniloquinoline by using conventional and microwave irradiation methods. By overall studying the microwave method is best when compared with conventional method, which gives good yield and time consumption is very less. The synthesized compounds have been characterized by IR, NMR and Mass spectrum which exactly matches the formation of desired product. These compounds are screened for antibacterial activities which show that the obtained results are potent antibacterial agents.

References

[1] "Microwaves in organic synthesis'' organic chemistry portal
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[3] Brickner,S J., M.R.Barbachyn,D.K.Hutchinson and P.R.Manninen,the first member of a completely new class of antibacterial agents for treatment of serious gram positive infections.
[4] Synthesis of dibenzenapthyridine by K.J.R. Prasad et al.