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SYNTHESIS, CHARACTERIZATION AND BIOLOGICAL EVALUATIONS OF SOME NEWER S-TRIAZINE BASED CHALCONE AND THEIR DERIVATIVES

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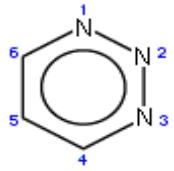
ABSTRACT

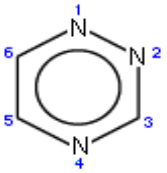
Triazines are The Chemical Species of Six-membered Heterocyclic Ring Compound with Three Nitrogen's Replacing Carbon-Hydrogen Units in The Benzene Ring Structure. 1,3,5-triazine Based Chalcones Have Been Prepared by The Claisen-Schmidt Condensation. Chalcones Have Characteristic 1, 3-diaryl-2-propen-1-one Backbone Skeleton. Changes in Their Aryl Rings Have Accessible a High Degree of Variety That Has Proven Useful For The Development of New Medicinal Agents with Improved Potency and Lesser Toxicity. The Structures of The Compounds were confirmed by Spectral Data (IR, 1H NMR and Mass spectroscopy). The Synthesized Compounds Were Studied for Their Antimicrobial Activity.

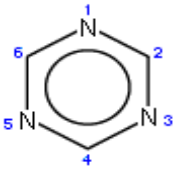
KEYWORDS: 1,3,5-Triazine, Cyanuric Chloride, Anti Microbial activity, Triazines Chalcones.

INTRODUCTION

Triazine structure is a heterocyclic ring, analogous to the six membered benzene ring with three carbons replaced by nitrogen's. The three isomers of triazine are distinguished from each other by their nitrogen atoms, and are referred to as 1,2,3- triazine, 1,2,4-triazines and 1,3,5-triazine or s-triazine. Triazines are weaker bases than pyridine. they have much weaker resonance energy than benzene, so nucliophilic substitution is preferred than electrophilic substitution (20 kcal/ mole and 49.8 kcal/mole) for 1,3,5-triazine and benzene respective The current project work is on 1,3,5,- triazine and its derivatives.

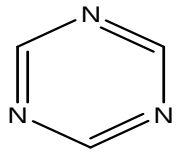

1,2,3-triazine


1,2,4-triazine


1,3,5-triazine
or
s-triazine

1,3,5,-triazines[2]

1,3,5-triazine, also called s-triazine, is an organic chemical compound with the formula $(HCN)_3$. It is a six-membered heterocyclic aromatic ring, one of several isomeric triazines. 1,3,5-triazine and its derivatives are useful in a various biological studies.


1,3,5-triazine

Synonyms: Sym-Triazine, s-Triazine, cyanidine, hydrogen cyanide trimer, vedita.

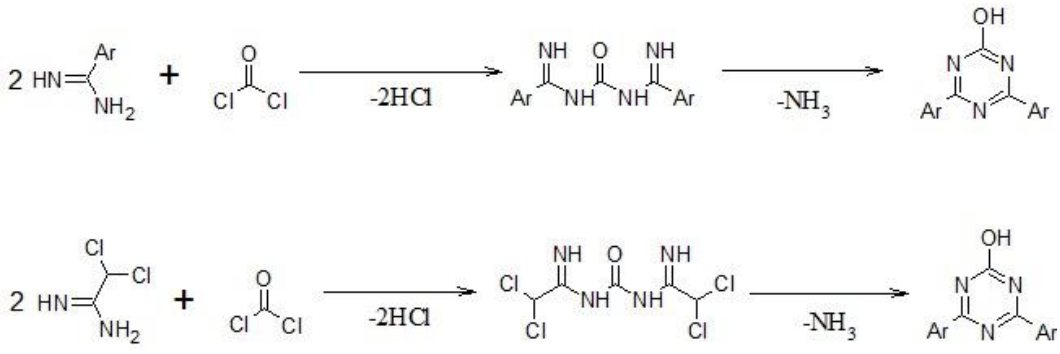
Properties

1,3,5-Triazine is a White crystalline solid with molecular formula $C_3H_3N_3$ and molar mass 81.08 g/ml. Melting Point 81-83°C. It has a polar molecular shape and Zero dipole moment.

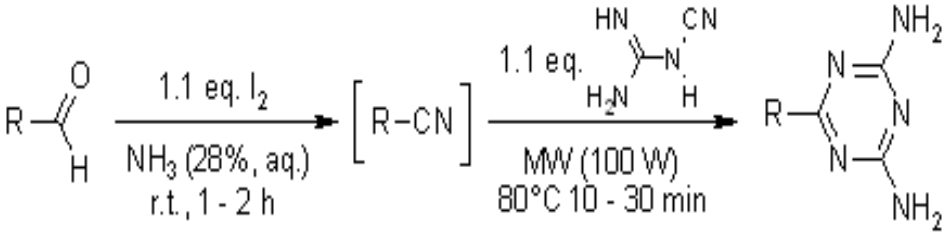
General methods of synthesis of 1,3,5-triazines

Pinner synthesis[3]

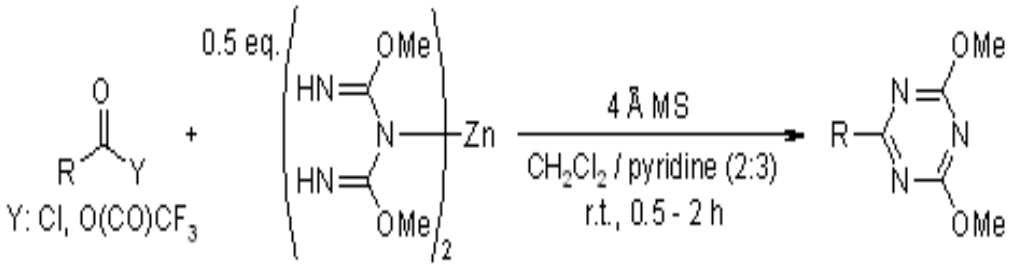
The **Pinner triazine synthesis** describes the preparation of 2-hydroxyl-4,6-diaryl-*s*-triazines by reaction of aryl amidines and phosgene via the intermediate of bisimidyl urea and is referred to as the pinner synthesis. The yields of the reaction depends on the temperature however, this reaction might not be suitable for aliphatic amidines. It has been reported that various other types of sym-triazines can be prepared from the thermal triamerization of amidine.



2. A series of primary alcohols and aldehydes were treated with iodine in ammonia water under microwave irradiation to give the intermediate nitriles, which without isolation underwent [2 + 3] cycloadditions with dicyandiamide and sodium azide to afford the corresponding triazines and tetrazoles in high yields.



3. Activated carboxylic acids react with a stoichiometric amount of zinc dimethyl imidodicarbonimidate in CH_2Cl_2-pyridine with molecular sieves to form 4,6-dimethoxy-1,3,5-triazines in high yields.



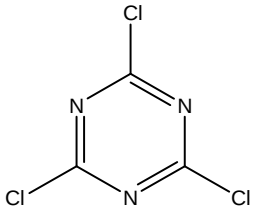
Applications of 1,3,5-triazine[4]

- As a reagent in organic synthesis, 1,3,5-triazine is used as the equivalent of hydrogen cyanide (HCN). Being a solid (vs, a gas for HCN), triazine is sometimes easier to handle in the laboratory. One application is in the gattermann reaction, used to attach the formyl group to aromatic substrates. It is a common reagent, and readily forms derivatives, which are used as pharmaceutical products, s well as herbicides, such as triazine.
- Triazines are basic structure of herbicides, like amitole, atrazine, cyanazine, simazine, trietazine, large volume of triazines is used in the manufacture of resin modifier such as melamine and benzo guanamine.
- Triazines are also useful as chromophore groups in colorants and chlorine attached in triazine compounds undergo nucleophile substitution reaction well with hydroxyl groups in cellulose fibres.
- Some triazine family compounds are used in pharmaceutical industry as coupling agent for the synthesis of peptide in solid phase as well as solution and as side chain of antibiotics.

- Triazine compounds are used in formulating bactericide and fungicide. They are used as preservatives in oil field applications, disinfectants, industrial deodorants, biocide in water treatment, bleaching agents, etc

Cyanuric chloride[5]

Cyanuric chloride is an organic compound with the formula $(NCCl)_3$. This white solid is the chlorinated derivative of 1,3,5-triazine. It is the trimer of cyanogen chloride. Cyanuric chloride is the main precursor to the popular but controversial herbicide atrazine. It's the trimer of cyanogen chloride.

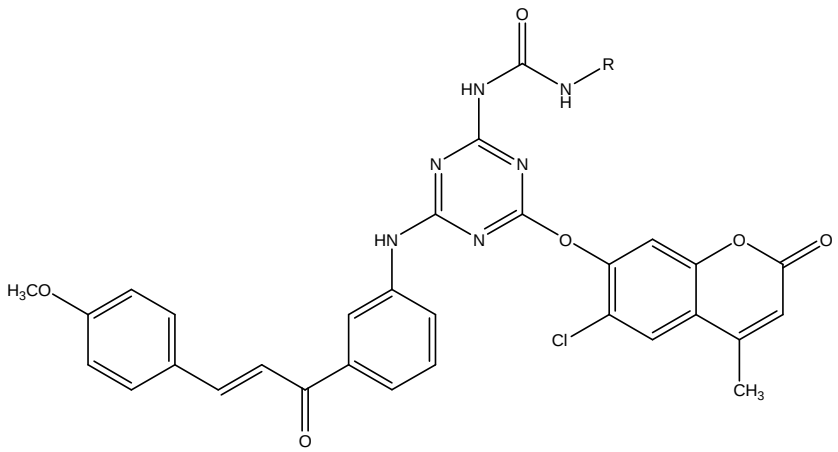


cyanuric chloride

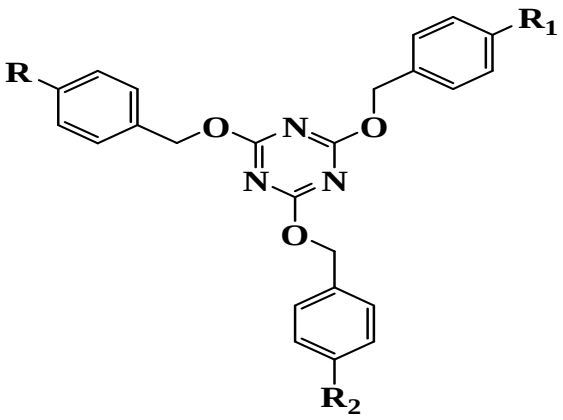
Synonyms: Trichloro triazines, s-triazine trichloride, cyanuryl chloride.

LITERATURE ON BIOLOGICAL ACITIVITY STUDIES OF DIFFERENT 1,3,5-TRIAZINE DERIVATIVE

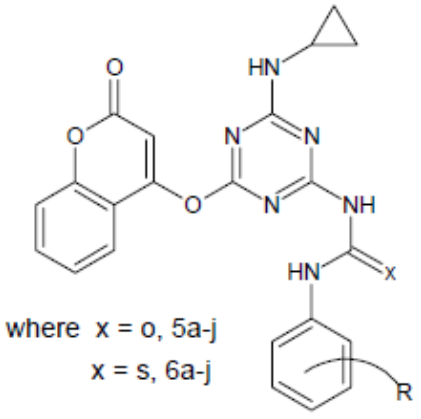
C. K. Naik and V. A. Desai et al[6] A new series of s-triazine derivatives, aryl amino s-triazine and aryl ureido s-triazine has been synthesized by the condensation of aryl amine and aryl ureido with 2- [4'- methyl - 6' - chloro -2*H*- chromen -2' -one-7'-oxy]-4-(3'- acetyl aminophenyl)-6-chloro s-triazine in acetone as a solvent and Potassium carbonate using as a neutralizing agent. The structures of all these compounds were confirmed on the basis of their analytical and spectral data1. The title compounds were characterized by IR, 1H NMR and elemental analysis and screening for their antibacterial activity.



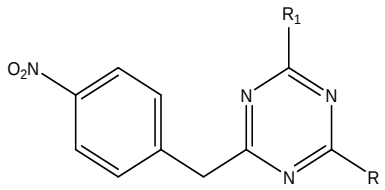
Srinivas et al[7] had synthesized various 2,4,6 –tri substituted s-triazines and screened for antibacterial activity against Gram-positive and Gram-negative organisms. These s-triazine derivatives displayed high in vitro antibacterial activities comparable to penicillin and streptomycin against tested microorganisms. Among them, compound displayed significant large activity against both Gram-positive and Gram-negative microorganisms.



Pankaj B. Kaswala,* Kishor et al[8] A series of urea and thiourea derivatives of s-triazine have been developed based on high yielding nucleophilic substitution of 2,4,6-trichloro-1,3,5-triazine by 4-hydroxy coumarin, cyclopropylamine and ammonia at suitable conditions. These were further treated with various substituted aryl isocyanate and aryl isothiocyanate. All the synthesized compounds were evaluated for their antibacterial activities against various Gram-positive and Gram-negative strains of bacteria. A few compounds showed good to superior *in vitro* antibacterial activity against *S.aureus, B.subtilis, E.coli* and *P.aeruginosa* respectively.



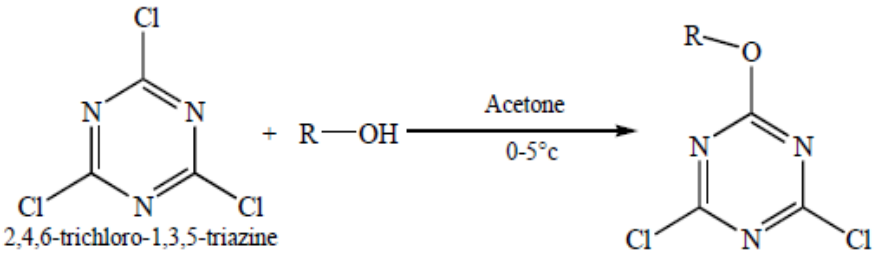
Agarwal et al.[9] was prepared a series of some new 2, 4, 6-trisubstituted-1, 3, 5-triazines and evaluated for their in vitro anti malarial activity against Plasmodium falciparum. Most of the compounds synthesized showed MIC in the range of 1–2 µg/mL.



EXPERIMENTAL (MATERIALS AND METHODS)

SCHEME FOR SYNTHESIS OF TRIAZINE DERIVATIVE

STEP 1



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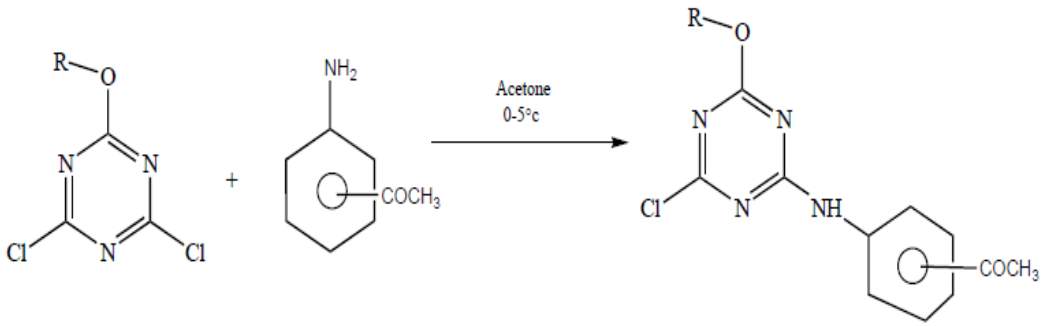

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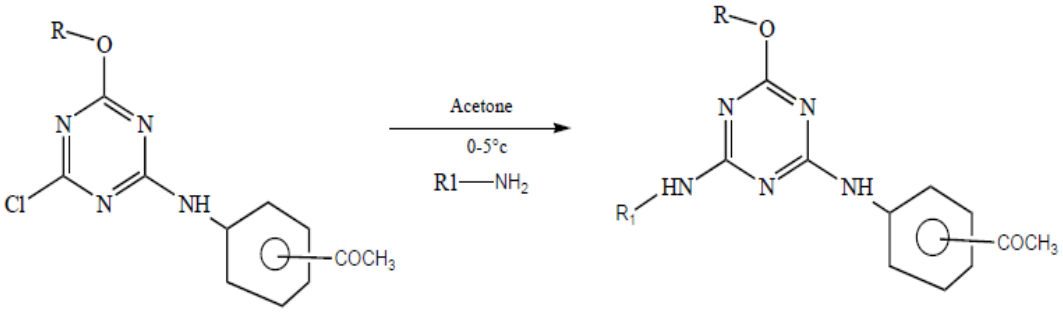

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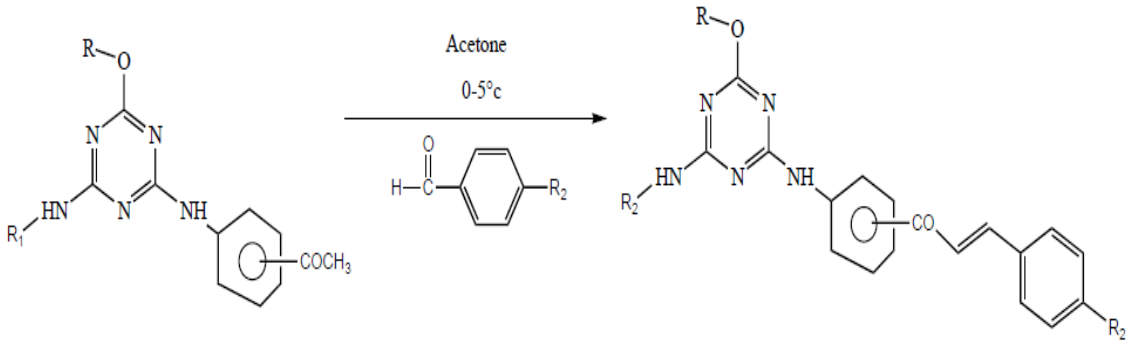
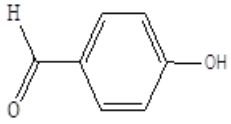
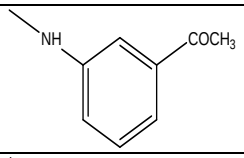
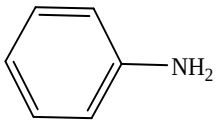
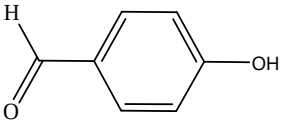
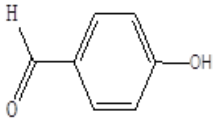
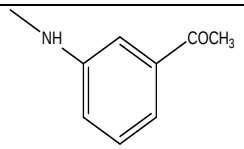
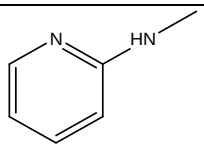
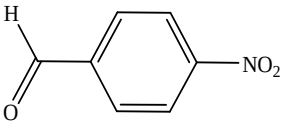
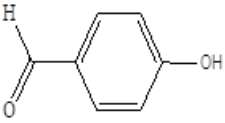
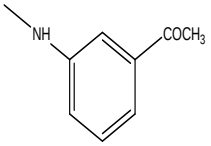
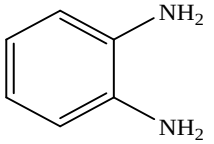
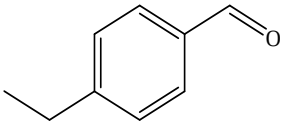
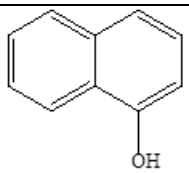
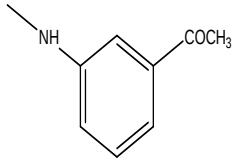
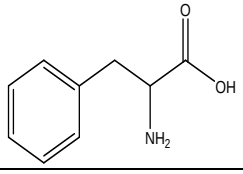
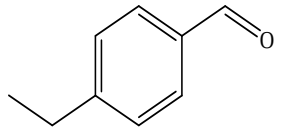
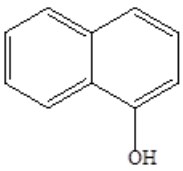
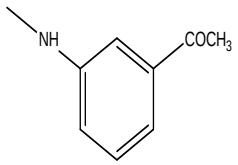
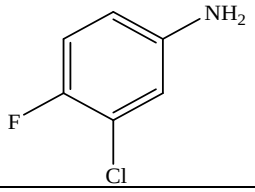
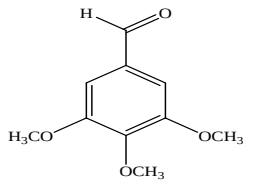
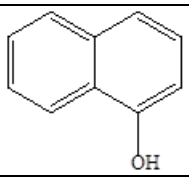
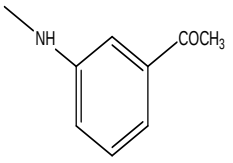
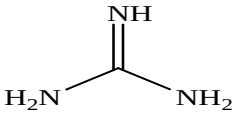
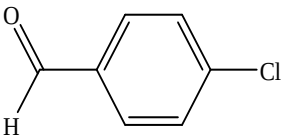


Table 1: Derivative Compounds.

S.NO	Code	R	R1	R2	R3
1.	A1				
2.	A2				
3.	A3				
4.	A4				
5.	A5				
6.	A6				

PROCEDURE FOR SYNTHESIS

Materials required

Cyanuric chloride, Acetone. Different amines and aldehydes, sodium carbonate, distilled water, ethanol

Apparatus used

Beakers, ice bath, magnetic stirrer, glass rod, heating mantle, pH paper, vaccum pump.

Step 1: synthesis of 2-(4,6-dichloro-1,3,5-triazin-2-yloxy)benzaldehyde

2-hydroxy compound was added slowly to the solution of 0.01 mol cyanuric chloride in 50 ml acetone with constant stirring for in 4 hours at 0°C, during the reaction sodium carbonate solution was added to neutralize to HCl evolved and Reaction was monitored by TLC (ethyl alcohol 4 ml + Toluene 6ml). After completion of reaction, the reaction mass was quenched re-in crushed ice and the solid obtained was filtered, washed with water and crystallized in ethanol.

Step 2: synthesis of 2-(6-cholro, 1,3,5-triazin- 2yloxy), 4-(3-acetyl amino phenyl), 6-chloro s-triazine

0.01 mole substitution--amino acetophenone was dissolved in 25 ml of acetone, which was added slowly to 0.01 mole compound (VI) in 50 ml acetone at such a rate that the temperature of the reaction mixture did not rise above 28 °C and addition completed in 4 to 5 hours. During the reaction proceed sodium carbonate solution was added to neutralize to HCl evolved. Reaction was monitored by TLC (ethyl alcohol 4 ml + Toluene 6ml). After completion of reaction, the reaction mass was quenched in crushed ice and the solid obtained was filtered, washed with water and crystallized in ethanol.

Step 3: synthesis of 2-(1,3,5-triazin- 2yloxy), 4-(3-acetyl amino phenyl), 6-aryl amine s-triazine

A mixture of 0.01 mole substituted amine and 0.01 mole compound (VIII) was reflux in 50 ml acetone. During the reaction proceed sodium carbonate solution was added to neutralize to HCl evolved. Reaction was monitored by TLC (ethyl alcohol 4 ml + Toluene 6ml). After completion of reaction, the reaction mass was cool to room temperature and quenched in crushed ice and the solid obtained was filtered, washed with water and crystallized in alcohol to get product.

Step 4: synthesis of 2-(1,3,5-triazin- 2yloxy), 4-[3'- {3'' (-4''' - methoxy phenyl) - 2'' - propane - 1''-one}] phenyl amino-6-arylamino-S-triazine

Above compound 0.01 mole was dissolved in 50 ml methanol and 25 % KOH solution was added to it. Substituted compounds added under constant stirring at room temperature. The

reaction mixture was poured into crushed ice and neutralize with HCl. Compound is separated out was filtered, washed with water and crystallized from alcohol.

RESULT AND DISCUSSION

Table 2: Characterization Data of The Synthesized Derivatives.

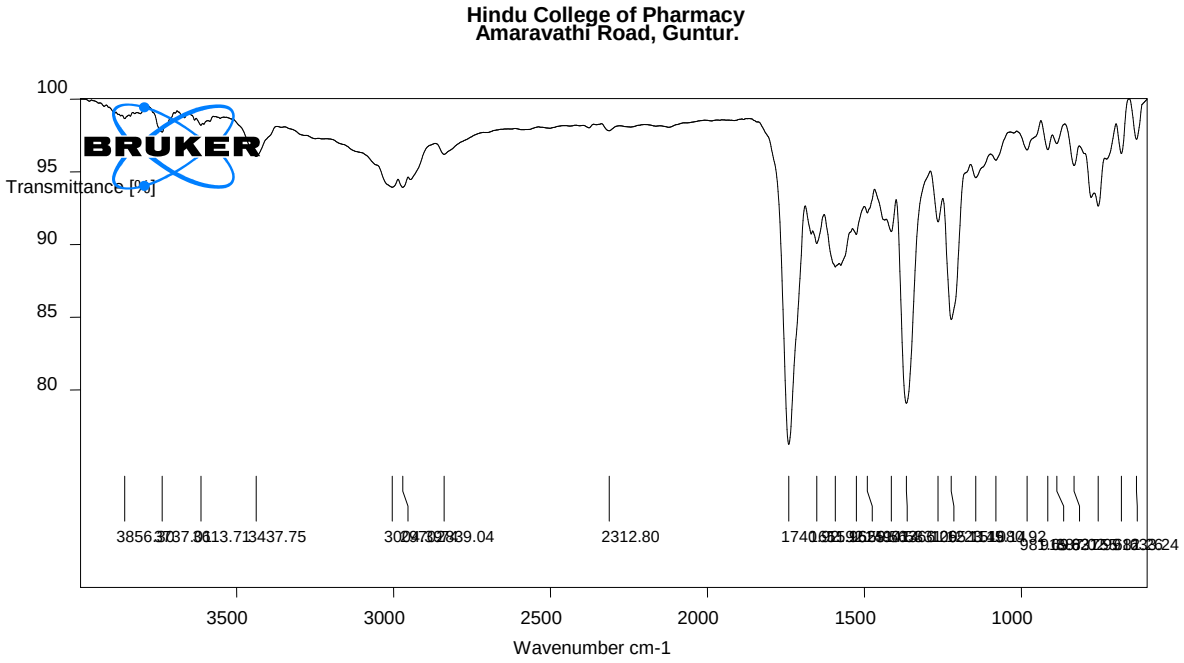
Compound code	Mol. Formula	Mol. Weight (g/mole)	M.P (°C)	% yield	Rf value*
A1	C30H26N2O4	478.54	225-230°C	61.77	0.81
A2	C31H28N2O4	492.57	240-243°C	62.21	0.84
A3	C36H29N5O2	563.65	250-255°C	57.03	0.78
A4	C35H28N6O3	580.64	225-230°C	61.56	0.83
A5	C35H25ClFN5O3	618.06	253-260°C	54.87	0.74
A6	C30H25N7O3	531.56	215-225°C	57.78	0.80

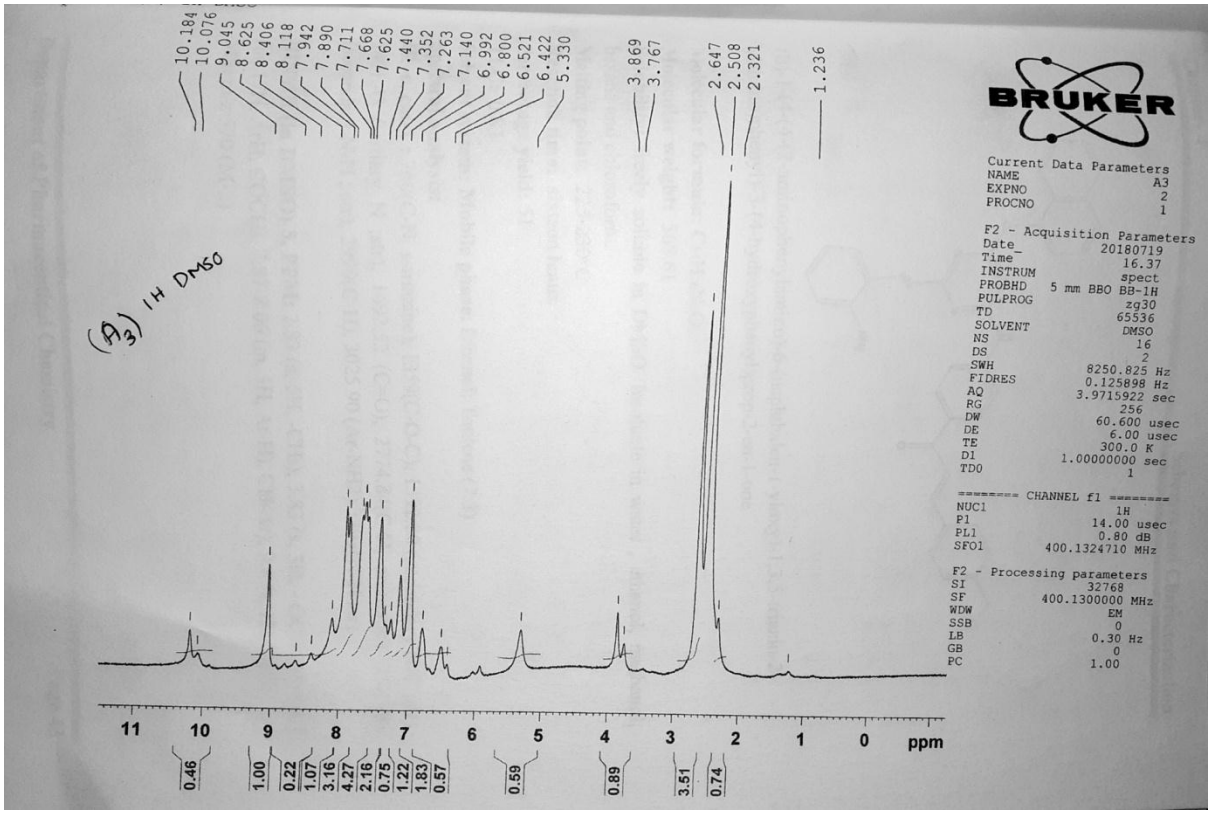
*Mobile phase = Ethanol + Toluene (7:3)

Table 3: Spectral Data of The Synthesized Derivatives.

Comp. code	Mol. formula	Spectral data
A1	C30H26N2O4	**IR υ, cm⁻¹:** 3131.66 (C-H, aromatic), 1730 (C=O), 808 (C-N, s-triazine), 3429(N-H str), 1340(C-O-C), 825(C-N), 2990(C-H), 1275.02 (secondary N-C ;str), 1321.43 (tertiary N ;str), 2835 (secondary N-H ; str). **¹HNMR (in DMSO) δ, PPM:** 2.83 (s, 6H, -CH3), 3.83 (s, 3H, - OCH3), 6.8-8.1 (m, 17H, ArH, COCH3, CH-Ar), 9.8 (s, 1H, NH),
A2	C31H28N2O4	**IR υ, cm⁻¹:** 2985.07 (C-H, aromatic), 1650 (C=O), 774.78 (C-N, s-triazine), 3426(N-H str), 1340(C-O-C), 826(C-N), 2990(C-H), 1367.05 (secondary N-C ;str), 1438.43 (tertiary N ;str), 2835.26 (secondary N-H ; str). **¹HNMR (in DMSO) δ, PPM:** 2.83 (s, 6H, -CH3), 3.83 (s, 3H, - OCH3), 6.8-8.1 (m, 17H, ArH, COCH, 7.87-8.06 (m, 5H, Ar-H), CH-Ar), 9.8 (s, 1H, NH).
A3	C36H29N5O2	**IR υ, cm⁻¹:** 2985.07 (C-H, aromatic), 1653.27 (C=O), 755.73 (C-N, s-triazine), 3433(N-H str), 1264(C-O-C), 826(C-N), 2990(C-H), 1366.05 (Ar-NO2), 1412 (secondary N-C ;str), 1491.43 (tertiary N ;str), 2835.81 (secondary N-H ; str). **¹HNMR (in DMSO) δ, PPM:** 2.83 (s, 6H, -CH3), 3.83 (s, 3H, - OCH3), 6.8-8.1 (m, 17H, ArH, COCH, 7.87-8.06 (m, 5H, Ar-H), CH-Ar), 9.8 (s, 1H, NH).
A4	C35H28N6O3	**IR υ, cm⁻¹:** 2774.84(C-H, aromatic), 1692.52 (C=O), 760(C-N, s-triazine), 3393(N-H str), 1359(C-O-C), 2990(C-H), 3025.90 (Ar-NH2), 1392.80 (secondary N-C ;str), 1491.43 (tertiary N ;str), 2835.81 (secondary N-H ; str).

		¹HNMR (in DMSO) δ, PPM: 2.83 (s, 6H, -CH3), 3.83 (s, 3H, - OCH3), 6.8-8.1 (m, 17H, ArH, COCH, 7.87-8.06 (m, 5H, Ar-H), CH-Ar), 9.8 (s, 1H, NH). **Mass** : m/z 570.8 (M^{+2})
A5	C35H25ClFN5O3	**IR υ, cm⁻¹: :** 2779.11(C-H, aromatic), 1699.10 (C=O), 794(C-Cl, s-triazine), 3393.83(N-H str), 1359(C-O-C), 2990(C-H), 3607.19 (Ar-OH), 1349.11 (secondary N-C ;str), 1851.44 (tertiary N ;str), 3076.43 (secondary N-H ; str). **¹HNMR (in DMSO) δ, PPM:** 2.83 (s, 6H, -CH3), 3.83 (s, 3H, - OCH3), 6.8-8.1 (m, 17H, ArH, COCH, 7.87-8.06 (m, 5H, Ar-H), CH-Ar), 9.8 (s, 1H, NH).
A6	C30H25N7O3	**IR υ, cm⁻¹:** 2746.91(C-H, aromatic), 1678.35 (C=O), 722(C-Cl, s-triazine), 3393.83(N-H str), 1228(C-O-C), 2990(C-H), 3614.89 (Ar-OH), 1321.23 (secondary N-C ;str), 3056.30 (secondary N-H ; str). **¹HNMR (in DMSO) δ, PPM:** 2.83 (s, 6H, -CH3), 3.83 (s, 3H, - OCH3), 6.8-8.1 (m, 17H, ArH, COCH, 7.87-8.06 (m, 5H, Ar-H), CH-Ar), 9.8 (s, 1H, NH).

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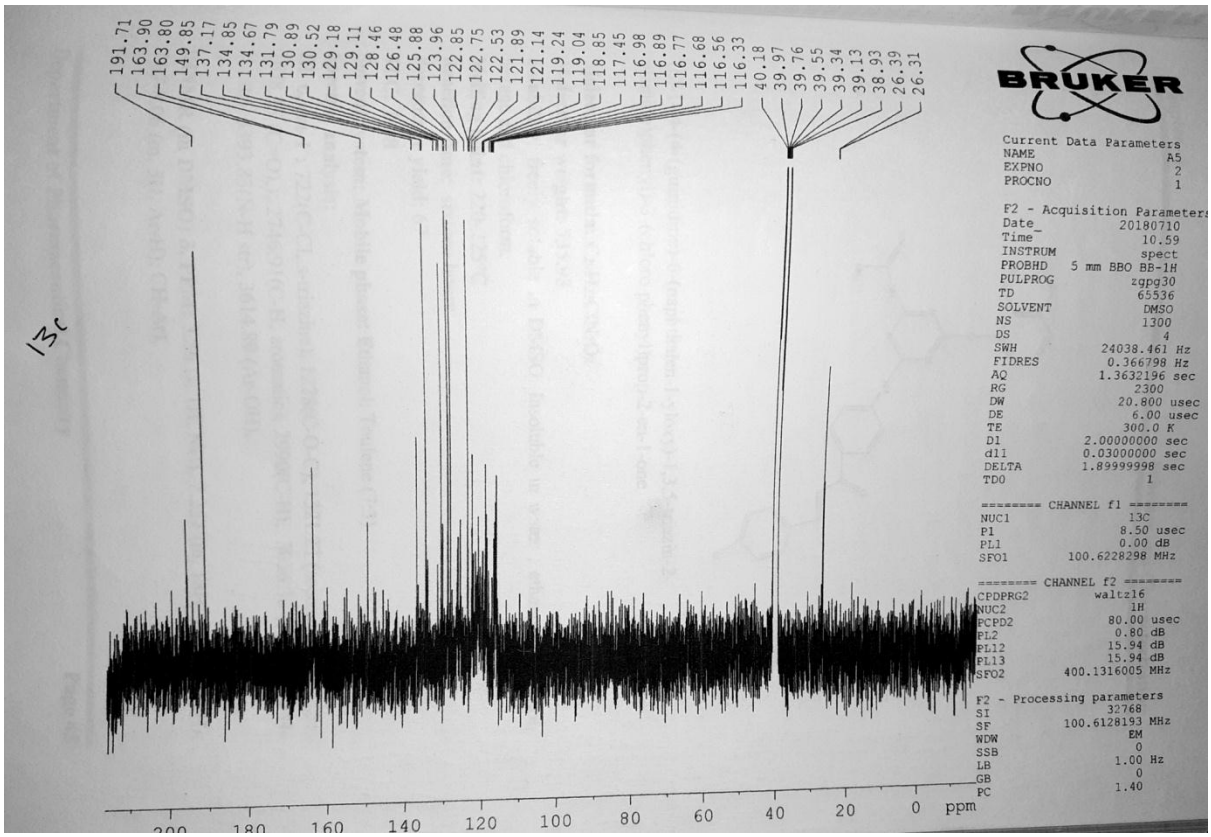
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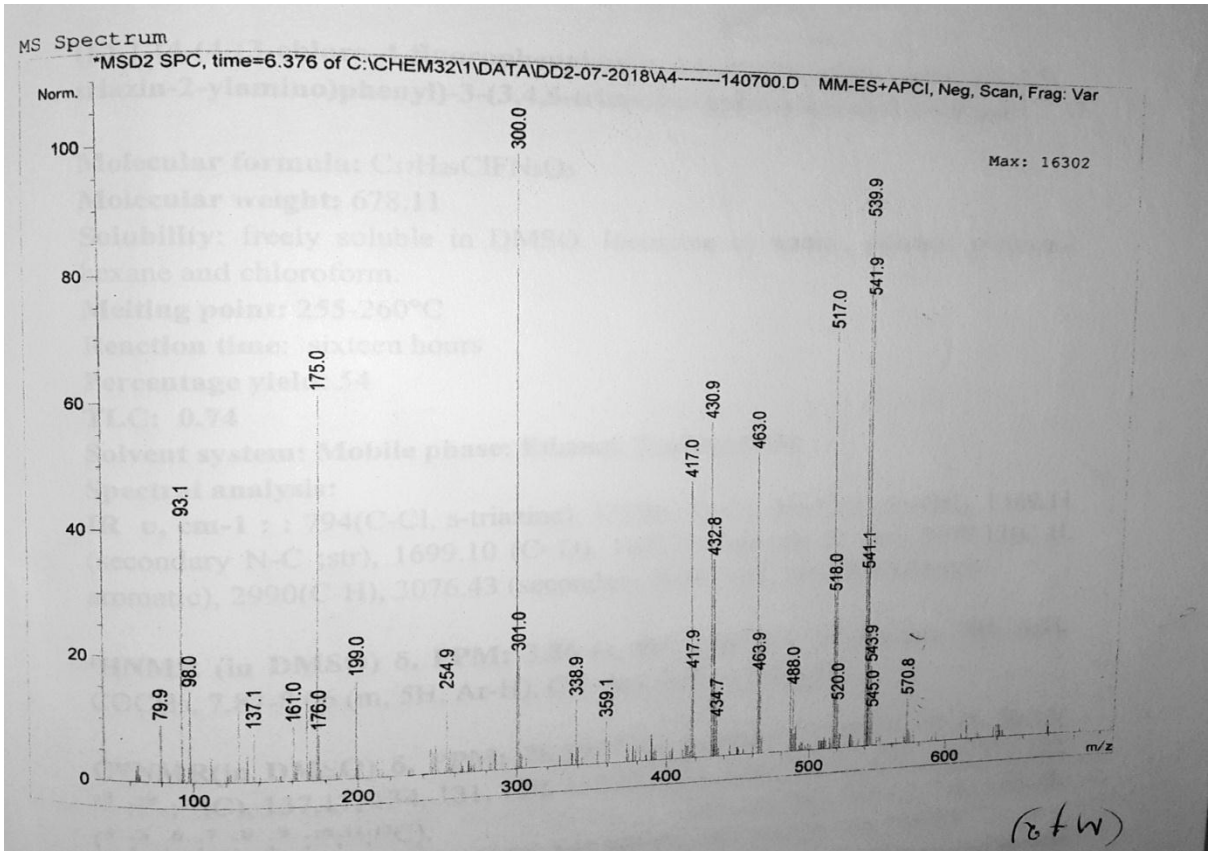

Table 3: Zone of Inhibition (In Mm) Obtained On Bacteria.

S.no	Compound code	Gram +ve				Gram –ve	
		S.aureus		B.pimilis		E.coli	
		500 µg/ml	1000 µg/ml	500 µg/ml	1000 µg/ml	500 µg/ml	1000 µg/ml
1.	A1	0	0	0	0	1	4
2.	A2	0	0	0	0	0	0
3.	A3	0	0	0	0	4	8
4.	A4	0	0	0	0	5	8
5.	A5	0	0	0	0	0	0
6.	A6	0	0	0	0	0	0
control	DMSO	0		0		0	
Standard	Streptomycin	6		4		8	

(*) significant zone of inhibition bore size-8 or 10mm

A total of 6 compounds were synthesized from scheme 1 were screened for anti bacterial activity at concentrations 50 and 100 µg/ml. among the all compounds, A3 and A4 were found to have moderate activity against gram –ve E. coli. Remaining all compounds were found to have less or no activity against both gram +ve and gram –ve bacteria, compared to the standard streptomycin.

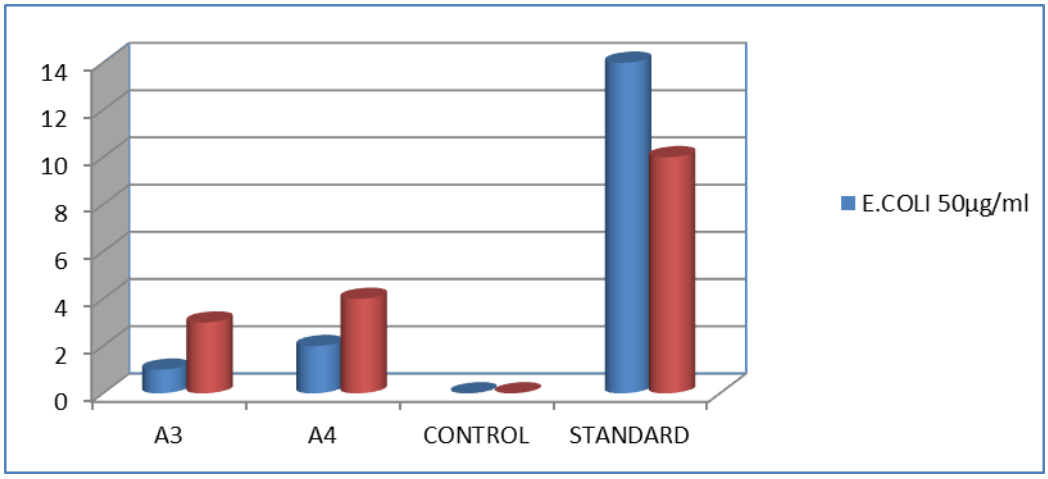

Figure 1: Graph Measurement of Anti Bacterial Activity.

Table 4: Zone Of Inhibition (In Mm) Obtained On Fungi.

S.no	Compound code	Aspergillus niger		Penicillum notatum	
		500 µg/ml	1000 µg/ml	500 µg/ml	1000 µg/ml
1.	A1	0	0	1	4
2.	A2	0	0	0	0
3.	A3	0	0	1	4
4.	A4	0	0	3	5*
5.	A5	0	0	0	0
6.	A6	0	0	0	0
control	DMSO	0	0	0	0
standard	Streptomycin	14*		10*	

(*) significant zone of inhibition bore size – 8 or 10 mm

The synthesized derivative compounds of 1,3,5-triazines were evaluated for anti fungal activity of cup plate method at the concentration of 50 µg/ml and 100 µg/ml using aspergillus nigur and pencillum notatum. Standard used was miconazole nitrate and control was DMF.

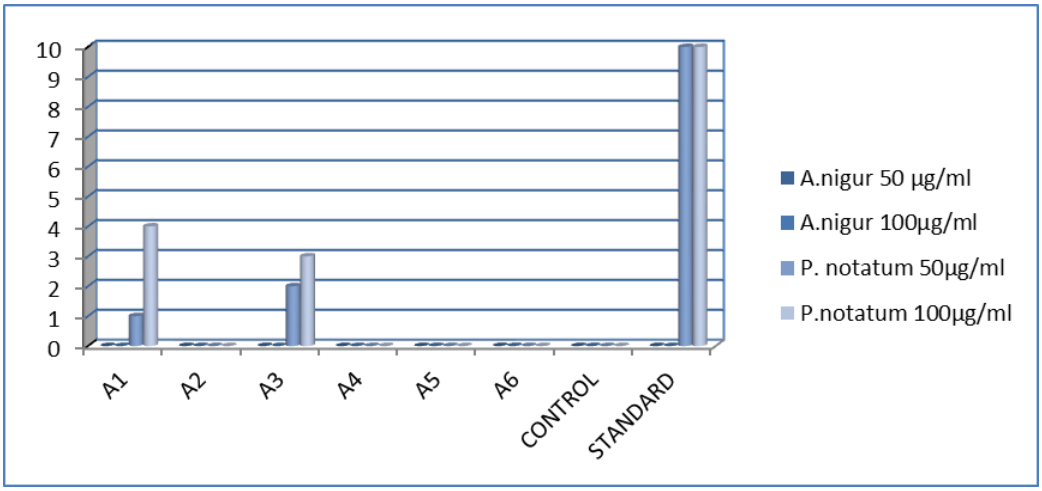

Figure 2: Graph Measurement of Anti Fungal Activity.

CONCLUSION

1,3,5-triazine is one of the oldest heterocyclic compound available. Because of its low cost and easily availability. Some dyes, lubricants and reagents derived from 1,3,5-triazine are already available in market. In this work, *1,3,5*--triazine based chalcone were successfully synthesized using Claisen-Schmidt condensation method.1,3,5-triazine based chalcone provided a versatile synthetic approach for the synthesis of differently bioactive substituted triazine chalcones. The synthetic yields of the generated products ranged from 69 to 75 % and their structures were established by spectral data (IR, NMR, and MS). The compounds N2, N4, N6- substituted- 1,3,5-triazines were synthesized from cyanuric chloride and characterized by spectral studies. The compounds were evaluated by antimicrobial activity. The results obtained indicate that a majority of compounds show only moderate insignificant activity.

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