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Synthesis, Characterization and Antibacterial activity of halogen dcerivatives1, 3, 4-thiadiazole

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Abstract

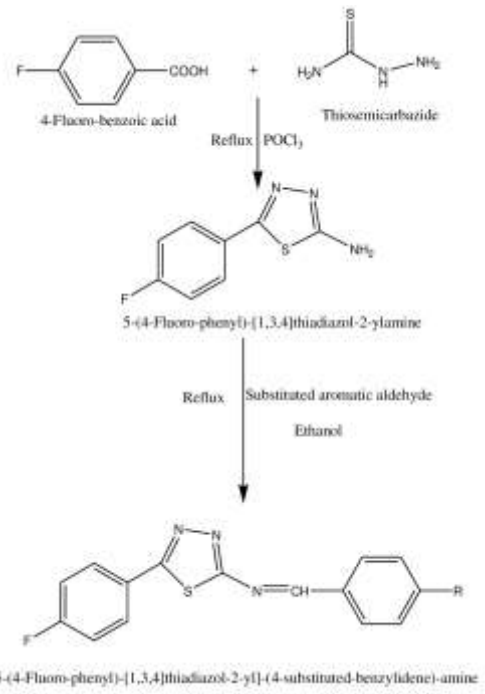
1,3,4-thiadiazole compounds are under come the category of heterocyclic compounds. Some new 1,3,4-thiadiazole compounds has been synthesize to use halogens like chlorine, fluorine etc. Initially 4-flouro benzoic acid and thiosemicabazide has been taken in the presence of $POCl_3$. An intermediate product was yield named 5-(4-Fluoro-phenyl)-[1,3,4]thiadiazol-2-ylamine then with the help of halogens derivatives two coumpound synthesized i.e. 4-Fluoro-benzylidene)-[5-(4-fluoro-phenyl)-[1,3,4]thiadiazol-2-yl]-amine and 4-Chloro-benzylidene)-[5-(4-fluoro-phenyl)-[1,3,4]thiadiazol-2-yl]-amine. Both compounds characterized by IR, Mass & NMR then they were send to evaluate antibacterial activity.

Key-words: Thiadiazole, Antibacterial, Halogens

Introduction

Heterocyclic moieties can be found in tremendous number of healing blends which show shifts type regular activity. [1] Numerous medications containing 1,3,4-thiadiazole (1,3,4Thiz) focus like acetazolamide, butazolamide, sulfamethazole are open in feature. In expansion different analogs have seen to be utilized as concealing, pesticides, oils and driving polymers [2]

1, 3, 4-thiadiazole (1,3,4Thiz) is a five part ring heterocyclic chromophore containing one sulfur molecule and two nitrogen heteroatom. It's have four type of isomer as 1,2,3-thiadiazole, 1,2,5-thiadiazole, 1,2,4-thiadiazole, 1,3,4-thiadiazoles but 1, 3, 4-thiadiazole most potent compound for different type biological activity like antimicrobial activity [3-7].



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Material and Methods

Synthesis of 5-(4-Fluoro-phenyl)-[1,3,4]thiadiazol-2-ylamine (Intermediate Compound)

An equimolar amount of 4-Fluoro -benzoic acid and Thiosemicarbazide in $POCl_3$ (10ml) was included and refluxed for 5 hours. The blend was cooled, poured on to squashed ice. The arrangement was washed, sifted and recrystallized from ethanol.

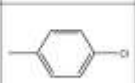
1. Synthesis of (4-Fluoro-benzylidene)-[5-(4-fluoro-phenyl)-[1,3,4]thiadiazol-2-yl]-amine

5-(4-Fluoro-phenyl)-[1, 3, 4] thiadiazol-2-ylamine with 4-Flurobenzaldehyde in 15 ml ethanol was refluxed for 3 hours. The mixture was cooled, poured on to crushed ice. The arrangement was washed, sifted and recrystallized from ethanol.

2. Synthesis of (4-Chloro-benzylidene)-[5-(4-fluoro-phenyl)-[1,3,4]thiadiazol-2-yl]-amine

5-(4-Fluoro-phenyl)-[1,3,4]thiadiazol-2-ylamine with 4 -chlorobenzaldehyde in 15 ml ethanol was refluxed for 3 hour. The mixture was cooled, poured on to crushed ice. The arrangement was washed, sifted and recrystallized from ethanol.

Table 1: Physiochemical properties of the synthesized derivative

S. No.	Compounds	R	Molecular formula	Mol. Weight
1.	(4-Fluoro-benzylidene)-[5-(4-fluoro-phenyl)[1,3,4]thiadiazol -2-yl] –amine (C)		$C_{15}H_9F_2N_3S$	301.31
2.	(4-Chloro-benzylidene)-[5-(4-fluoro-phenyl)-[1,3,4]thiadiazol-2-yl]-amine (D)		$C_{15}H_9ClFN_3S$	317.77

Results and Discussion

Compounds have been synthesized by given scheme. Synthesized compounds Characterized using FTIR, ^{1}H-NMR and mass spectroscopy and Physiochemical properties of all synthesized compounds were determined. All compounds were found potent in antibacterial activity.

A. Reagents and solvents

All chemical used in laboratory work was proceured from S.D. Fine Chem Ltd, E. Merck India Ltd., CDH, and Sigma Aldrich Ltd. Liquid for solution and reagents were of LR grade. The silica gel G (60-120 mesh) utilized for systematic chromatography (TLC) was acquired from E. Merck India Ltd.

The dissolvable framework utilized for TLC: Benzene: Acetone (3:1)

B. Instruments/Equipments

Borosil glass ware use in the laboratory. The analysis of synthesized compounds was carried out using various instruments/equipments which are listed below:

Infra Red spectra –

Infra Red spectra FTIR spectra of compounds were recorded in Agilent Cary FTIR spectrophotometer

Proton magnetic resonance spectra (**Proton - NMR)-**

Proton – NMR spectra of compounds were determined Bruker 400 MHz instrument in tetramethylsilane [$(CH_3)_4Si$] (TMS) as internal standard

Determination of melting point

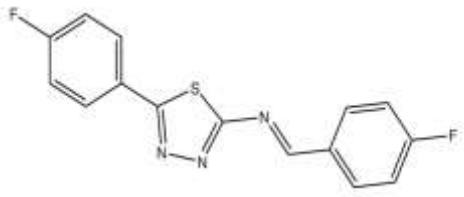
Melting points were noted by the open capillary method and were uncorrected.

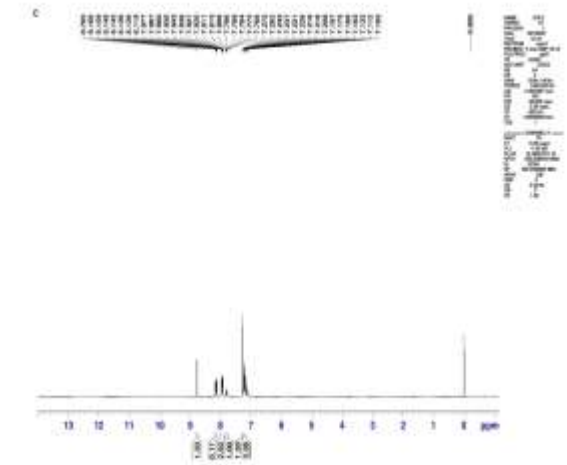
TLC of compounds –

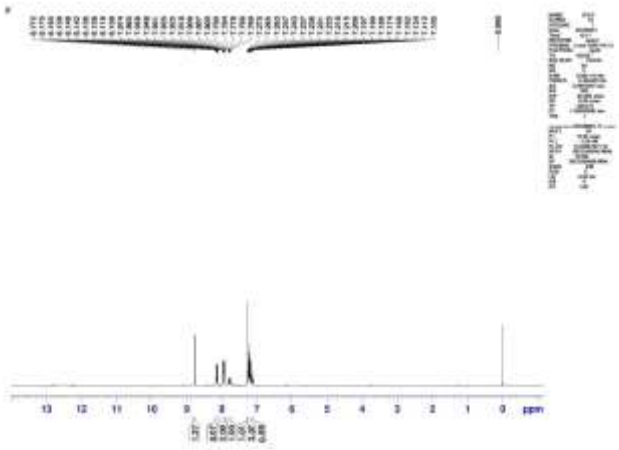
Silica gel G covered glass plate use for the thin layer chromatography assurance. The thickness of covering is about 0.3 mm on recently cleaned flimsy layer chromatography plates of 20x5 cm utilizing spreader. For the initiation of TLC plates were set in sight-seeing oven at 105 °c for 30 min. The sample spotting was done just above 2 cm above from lower edge of the TLC plate. The portable stage was chosen by the extremity of compound Benzene: Acetone 3:1. Iodine chamber and UV lights were utilized for representation of spots.

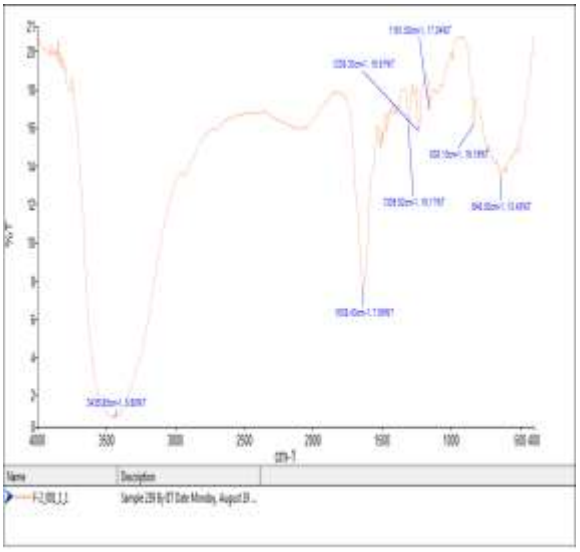
(4-Fluoro-benzylidene)-[5-(4-fluoro-phenyl)[1,3,4]thiadiazol -2-yl] –amine (C)

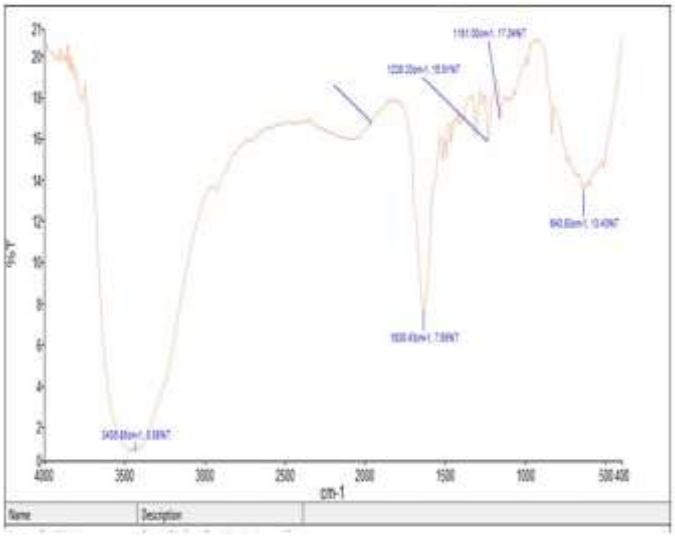
Mole. Formula: $C_{15}H_9F_2N_3S$,IR(KBr,cm^{-1}) υ: 2923.09(-H), 1629.33(C=C), 1563.41 (C=N) , 1106.38(C-F) ,^{1}HNMR: ($CDCl_3$400 MHz): δ 8.76(S,1H,=CH) , 8.16-7.105(m,8H,Ar-H).





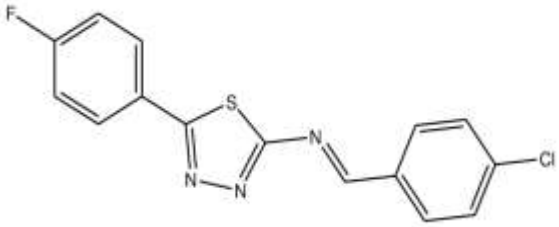






(4-Chloro-benzylidene)-[5-(4-fluoro-phenyl)-[1,3,4]thiadiazol-2-yl]-amine (D)

Mole. Formula: $C_{15}H_9ClFN_3S$,IR(KBr,cm^{-1}) υ: 2926.04(C-H), 1623.38(C=C), 1561.45 (C=N) , 1126.32(C-F) 623.43(C-Cl) ,^{1}HNMR:, ($CDCl_3$400 MHz): δ 8.77(S,1H,=CH) , 8.17-7.10(m,8H,Ar-H).



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