Iraqi National Journal of Chemistry

Journal homepage:
http://iqnjc.com/Default.aspx

Synthesis and Spectral Identification of N-[5-(pyridin-4-yl)-1,3,4-thiadiazol-2-yl] benzenesulfonamide

Maitham Mohamed Abdulridha
Technical institute of shatra
E-mail **: maitham008@yahoo.com**

ABSTRACT

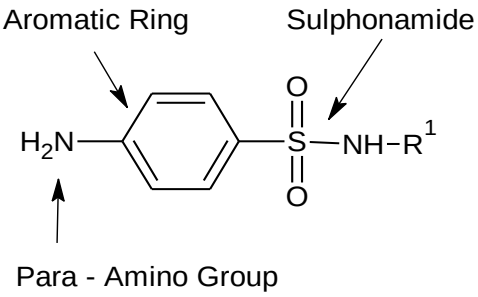
N-[5-(pyridin-4-yl)-1,3,4-thiadiazol-2-yl]benzenesulfonamide compounds are important due to have multi biological activity. In our present study sulpha compound derivative have been synthesized and identify by using the spectral methods of ^{1}HNMR , Mass ,IR, and C,H,N. The physical and chemical data suggested molecular and structural formula of synthesized compound.

Keywords: Identification.benzenesulfonamide.thiadiazole.

Introduction

Sulfonamides (sulfa drugs) were the first drugs largely employed and systematically used as preventive and chemotherapeutic agents against various diseases was first prepared in 1908 by Paul Gelmo as part of his dissertation for a doctoral degree from the Technische Hochsschule of Vienna, Austria as antibacterial [1].Sulfonamides represent an important class of medicinally important compounds which are extensively used as anti-inflammatory,antimicrobial,antifungal agents , anticancer, Urinary - tract infection [2].

The versatile biological actions of sulfa drugs is due to the presence of pharmacologically active sulfonamide group(.SO_2NH_2)and also due to the strategically placed amino group (-NH_2) at the para of the benzene ring is essential (R^1=H) ,Aromatic ring is essential, para-substitution is essential,sulphonamide group is essential ,ssulphonamide nitrogen must be primary or secondary R^2 can be varied . Structure-activity relationships of sulpha Drugs[3].



In present work , benzenesulfonyl chloride compound reacted with the primary amine (5-(pyridin-4-yl)-1,3,4-thiadiazol-2-amine) to produced Sulfonamide derivative. Thiadiazole is the heterocyclic compound five member ring which contain the nitrogen in different position as 1,3,4-thiadaizole,1,2,3-thiadiazole,1,2,4-thiadiazole,1,2,5-thiadiazole etc. The basic ring 1,3,4-thiadiazole is heterocyclic ring compound have many biological activities: such as antimicrobial activity, anti-inflammatory, anti-fungal, antibiotic, diuretic, anti-depressant [4].

Experimental work

First step : Synthesis of 5-(pyridin-4-yl)-1,3,4-thiadiazol-2-amine (A). A mixture of (0.01mo‹0.91gm) thiosemicarbazide and (0.01 mol, ١.٢٣gm) pyridine-4-carboxylic acid and (14 ml) of conc sulphuric acid in 100 ml of ethanol in two neck round bottom flask. fitted with a reflux condenser . The resultant reaction mixture was refluxed on a water bath for 4hr, then poured into crushed ice and neutralized with ammonia solution cautiously. A light yellow colored solid separates out. It was filtered off, washed with saturated sodium bicarbonate solution and water. and re-crystallized from ethanol. The purity of the compound was followed by TLC.The percentage of yield (71%,1.5 gm), melting point m.p.116 – 118°C.[5]

Second step:Synthesis of *N*-[5-(pyridin-4-yl)-1,3,4-thiadiazol-2-yl]benzenesulfonamide (B).
A mixture of(0.02 mol,3.5gm) synthesized compound (A) and appropriate (0.02 mol) benzenesulfonamide sulfonyl chloride in pyridine (15mL) was stirred at room temperature for 72 hrs. The mixture was evaporated under reduced pressure and the mixture was neutralized with 10% hydrochloric acid. The precipitate was filtered and washed with water and the resulting yellow crude product was purified by re-

crystallization from ethanol [6] . The purity of the compound was followed by TLC. The percentage of yield (68 %), melting point m.p. 216 – 218°C .

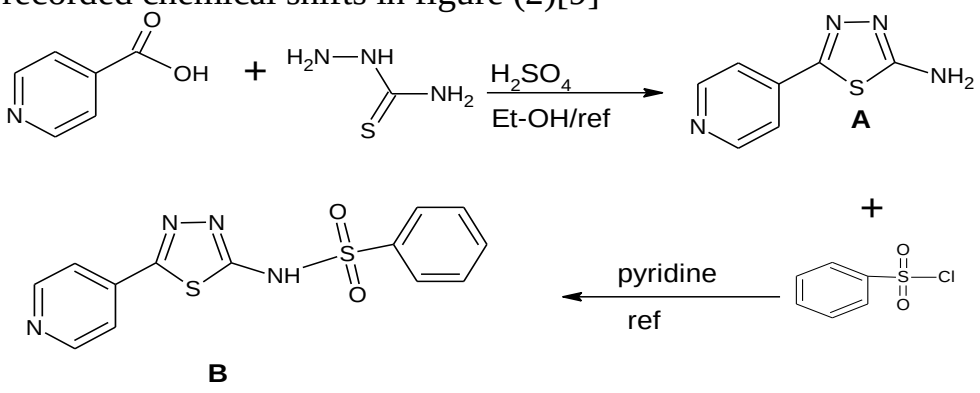
Results & Discussion:

The purity of synthesized compound A,B as showed in scheme No 1 were checked by TLC using silica Oxide as adsorbent . Molecular formula , melting points and yield tabulated in table (1)

The theoretical values of elemental analyses (C,H, N) examination as yield tabulated in table (1)were appeared a good agreement with the experimental values of C,H,N percentage in synthesized sulphonamide compound .[7] synthesized compound confirmed by IR, MASS , ^{1}H NMR spectra, . The calculated values were appeared agood agreement with the experimental values for the suggested formula .

The synthesized Sulphonamide compound(A)*N*-[5-(pyridin-4-yl)-1,3,4-thiadiazol-2-yl]benzenesulfonamide was identified by FTIR spectrum ,compound showed characteristic stretching absorption bands at 3309 cm^{-1} which were assigned to υN-H.and also showed characteristic absorption bands at 3129 cm^{-1} which were assigned to υ C-H of aromatic benzene ring .the following characteristic absorption bands 1613 cm^{-1}, corresponding to υC=N .and shows characteristic absorption bands at 1453 cm^{-1} due to υS=O . [8] as shown in figure (1)

The ¹H-NMR of the synthesized Sulphonamide compound *N*-[5-(pyridin-4-yl)-1,3,4-thiadiazol-2-yl]benzenesulfonamide showed the following peaks appeared: Singlet at 5.774 due to (, 1H, N-H), and showed Doublet at 6.778-7.012 due to Pyrridine Proton ring and also showed multiplet at 7.2–7.8 due to (4H, Ar-H) of benzene. The NMR Spectra give clear explain for support the composition of the synthesized Sulphonamide compound , therefore all the protons are at their expected region .The number of protons calculated from integration curves and the recorded chemical shifts in figure (2)[9]



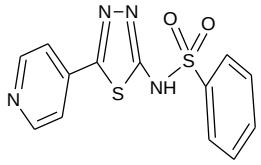
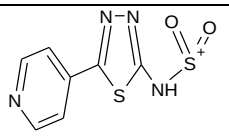
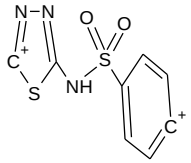
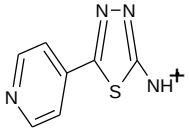
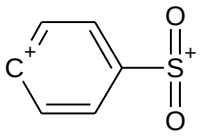
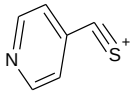
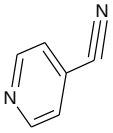
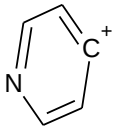
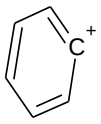
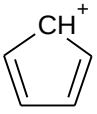
Scheme(1) of synthesized compounds A,B

Mass spectra:

The Mass spectrometer give strong support with extreme precision for the molecular weight value of the synthesized Sulphonamide compound (C). So by the Mass spectra the molecular formula have been predicted. The mass spectra of the Sulpha Compound showed a molecular ion peak fragment [M/Z] which is tabulated in the following table (2) . As showed in Figure (3). [10]

No.	M.F.	M. W. gm/mole	M.P°C	%Yield	Elemental analysis of synthesized Compound (A) C H N
A	$C_7H_6N_4S$	178	116-118	71%	%Theoretical Data 46.83 3.33 21.01
B	$C_{13}H_{10}N_4O_2S_2$	317	216-218	66 %	% practical D 46.96 3.40 20.05

Table 1. C H N , Molecular formula , Molecular weight ,melting point and yield of all synthesized compound .

Table 2 . The mass spectrum of compound (B)			
No	**Ion**	**Molecular Ion**	**Ion fragment structure**
1	$C_{13}H_9N_4O_2S_2$	317	
2	$C_7H_5N_4O_2S_2$	241	
3	$C_8H_5N_3O_2S_2$	239	
4	$C_7H_5N_4S$	177	
6	$C_6H_4O_2S$	141	
8	C_6H_4NS	122	
9	$C_6H_4N_2$	104	
12	C_5H_4N	78	
13	C_6H_4	76	
14	C_5H_4	64	

Conclusion

In the present work *N*-[5-(pyridin-4-yl)-1,3,4-thiadiazol-2-yl] benzenesulfonamide have synthesized (B) and characterized by spectral Data of IR,^{1}HNMR, Mass spectroscopic and by elemental analyses C,H,N .

According to the physical and chemical measurements of the prepared compound the chemical Sulphonamide Compound was successfully synthesized and expected have highly pharmacological therapeutic Activity . such as as antibacterial ,antimicrobial ,antitumor , anti-thyroid, anti-inflammatory, antiviral agents, antifungal agents, anticancer, diuretic , Urinary - tract infection.

تحضير و تشخيص المركب 1- (pyridin-4-YL) -5] -N،3،4-thiadiazol-2-YL] benzenesulfonamide

ميثم محمد عبدالرضا

المعهد التقني في الشطرة

البريد الالكتروني: maitham008@yahoo.com

الخلاصة

1- (pyridin-YL-4) -5] -N،3،4-thiadiazol-2-YL] benzenesulfonamide مركبات هامة نظرا لفعاليتها البيولوجية المتعددة. في الدراسة الحالية تم تحضير و تشخيص مشتقات السلفا باستخدام الطرق الطيفية للHNMR١، ، الأشعة تحت الحمراء، وC، H، N. وتشير البيانات الفيزيائية والكيميائية الصيغة الجزيئية والهيكلية للمركبات المحضرة.

Reference

1- Combs, Michael T. *Optimal analysis of sulfonamides from biological matrices using supercritical fluids*. Diss. Virginia Polytechnic Institute and State University, 1997.;P15.

2- Igwe, Christiana Nonye, and Uchechukwu Chris Okoro. "Synthesis, characterization, and evaluation for antibacterial and antifungal activities

of n-heteroaryl substituted benzene sulphonamides." *Organic Chemistry International* 2014 (2014).
3- Mohamed, Gehad G., and Carmen M. Sharaby. "Metal complexes of Schiff base derived from sulphametrole and o-vanilin: synthesis, spectral, thermal characterization and biological activity." *Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy* 66.4 (2007): 949-958.
4- Singh, Arvind K., Geeta Mishra, and Kshitiz Jyoti. "Review on Biological Activities of 1, 3, 4-Thiadiazole Derivatives." (2011).
5- Wang, Yao, et al. "5-(4-Pyridyl)-1, 3, 4-thiadiazol-2-amine." *Acta Crystallographica Section E: Structure Reports Online* 65.5 (2009): o1099-o1099.

6- Vikas, S., and S. Darbhamulla. "Synthesis, characterization and biological activities of substituted cinnoline culphonamides." *African health sciences*9.4 (2009).
7- P.Y. Pawar, B.Y. Mane, M.T. Salve and S.R. Bafana ; synthesis and anticonvulsant activity of n-substituted -7-hydroxy-4-methyl-2-oxa-quinoline derivatives; International Journal of Drug Research and Technology ; Int. J. Drug Res . Tech. 2013, Vol. 3 (3), 60-66.
8- Hussain, Zainab, et al. "Synthesis and characterization of Schiff's bases of sulfamethoxazole." *Organic and medicinal chemistry letters* 4.1 (2014): 1.
9- Hosseinzadeh, Nouraddin, et al. "Synthesis and antidiabetic evaluation of benzenesulfonamide derivatives." *Iranian Journal of Pharmaceutical Research* 12.2 (2013): 325-330.
10- Husain, Asif, et al. "New amides of sulphonamides: synthesis and biological evaluation." *Journal of the Chilean Chemical Society* 55.1 (2010):74-77.

Appendices:

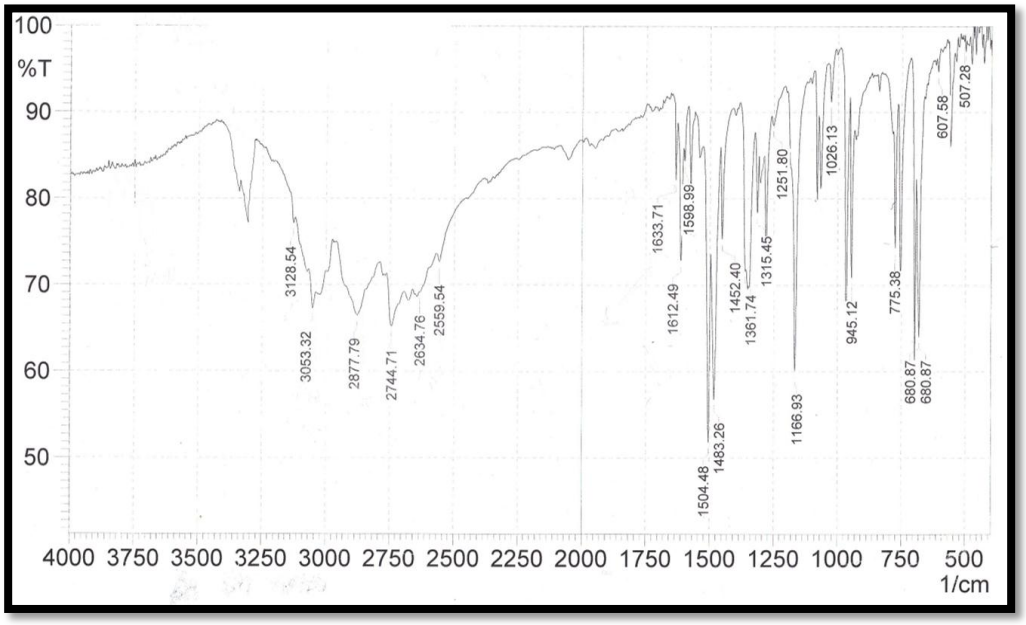


Figure (1) IR spectrum of the (B) compound cm^{-1}

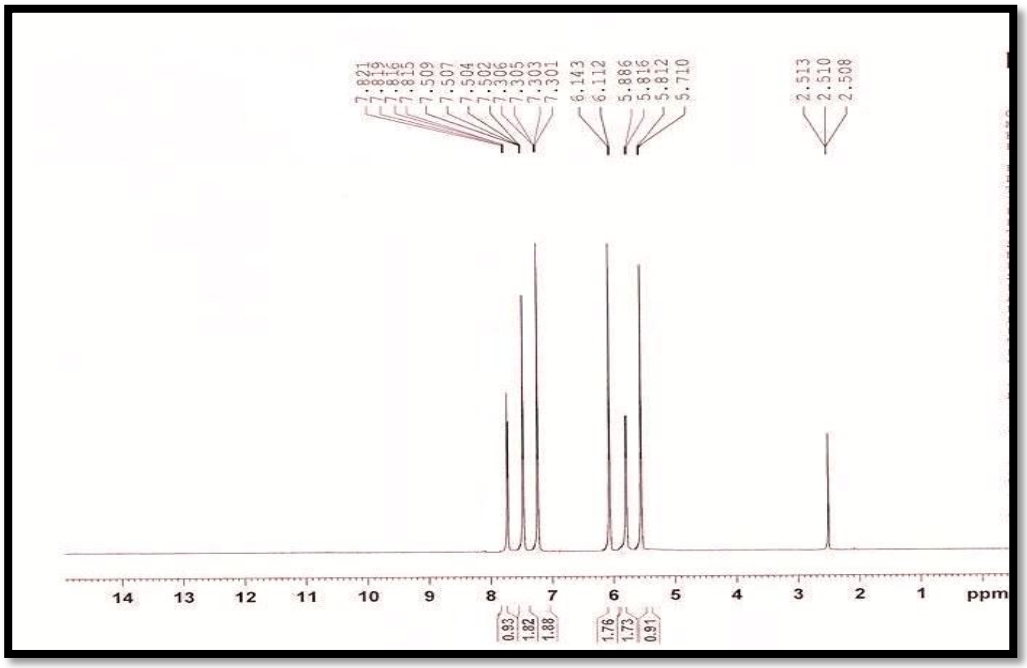


Figure (2) ^{1}H-NMR spectrum of the (B) compound cm-^{1}

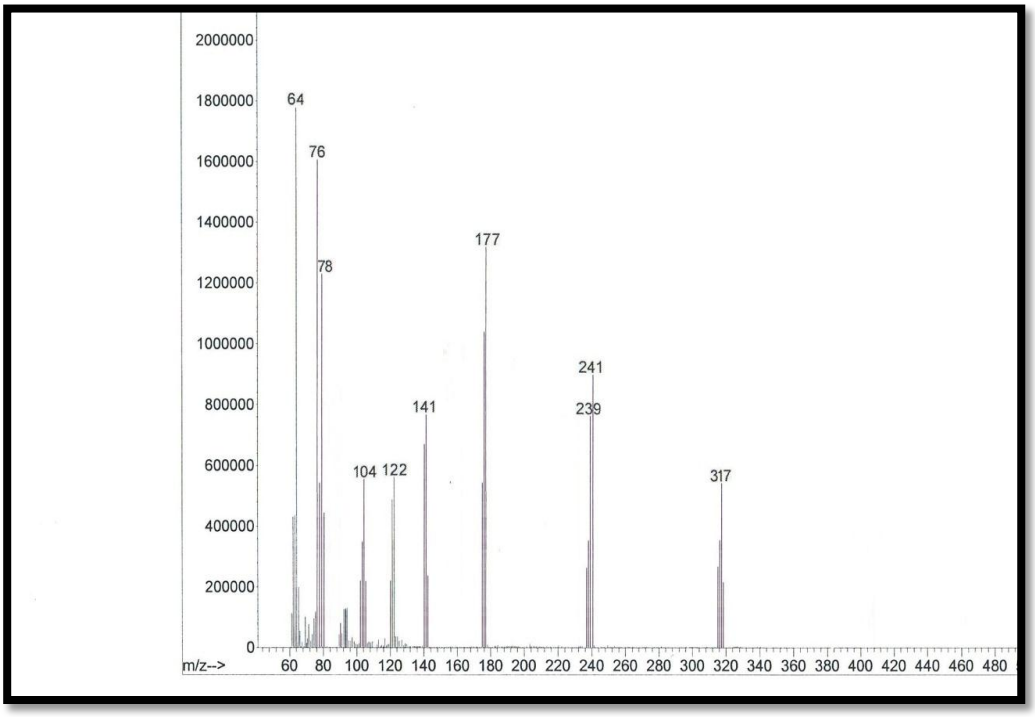


Figure (3) Mass spectrum of the (B) compound cm-^{1}