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Synthesis, Characterization and Fungal Evaluation against Fusarimoxysporium of New Azo-Pyrimidine Derivatives

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KEYWORDS
fungi,
derivative,
thiazo.

ABSTRACT:
Many researchers worked on mining important compounds from selected pharmaceutical plants and vitamins), especially those that included nitrogen atoms in their ring , in addition to manufacturing some of these compounds in the laboratory or laboratory to become therapeutic . Also, some natural nitrogen compounds were used as anti-corrosion materials ,dye materials, and permanent dyes and as antibacterial agents. Thiazoles are found in a variety of specialty products, and are often fused with benzene derivatives, so-called benzothiazoles. In addition to vitamin B1, several thiazole and pyrimidine derivatives were also prepared in this research, which represent azo-premidine compounds. These compounds also are identified and confirmed by many methods such as (FT- IR, 1HNMR, 13CNMR) spectra with evaluation against fungi.

INTRODUCTION

Azo compounds have been used for several past times and decades, and then developed at the present time in various fields of medicine, science and technology, giving results that are of great importance in life. Where monomeric azo compounds were used in many industries of dyes and cosmetics, and in the field of industry, they had a role Importantly, thiazolyl azo ligands have been widely used as chromogenic reagents, in addition to their uses in dyeing fabrics, polyester yarns, nylon, plastics, silk, and rubber. In the field of analytical chemistry, it exploited the characteristic color prevailing for this type of compounds and their complexes formed with metal ions in their aqueous solutions in spectral analyzes. Photography, where these dyes increase the sensitivity of imaging films, and in the field of organic diagnosis as reagents in the measurement of optical intensity, and in the field of medicine, they have been used as important drugs because of their role in inhibiting germs, and in medicine, Prontosil is the first azo dye to be used as an antibacterial., Researchers in congenital chemistry began to pay attention to rings that contain simple nitrogen atoms because they carry important doublets in electronic donation, as the science of chemistry

1. EXPERIMENTAL AND METHODS

Compound (Z1):
This compound prepared by dizo reaction through two steps in solvation amine with (HCl) and (NaNO2),then mixing with alkali solution of coupling compound, to yield product (Z1) ($C_{13}H_{11}N_7O_2S_2$) yield: Dark Orange (50%) m.p. (184-186) ^{0}C.

Synthesis of (Z2) :[2-chloro-N-(4-(N-pyrimidin-2-ylsulfamoyl)-2-(thiazol-2yl-diazenyl) phenyl) acetamide]
A mixture of Azo comp.(Z1) (0.001 mole, 0.361gm)and tri ethylamine (0.15ml) in DMF, Chloro acetyl Chloride (0.001 mole, 0.07ml) was added drop-wise. The reaction mixture was stirred for (5hrs.)at room temperature. After completion of the reaction, as indicated by TLC, the solvent was evaporatedat the end of reaction. Finally, the precipitate was dried and re-crystallized from absolute ethanol (Mkhaiber & Jarallah, 2022). ,(Z2) ($C_{15}H_{12}ClN_7O_3S_2$) yield: Light orange 53%, m.p.(177-179) ^{0}C, R_f (0.88) (Benz: MeOH, 2: 3).

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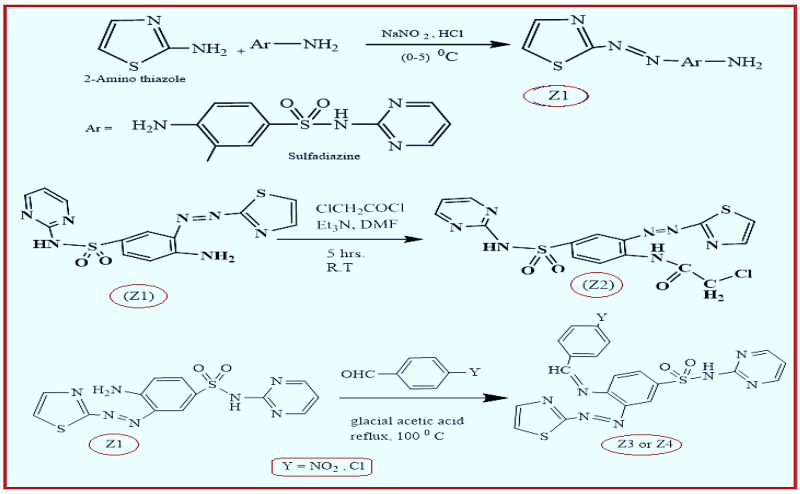
Synthesis of (Z3) [4-((Z)-4-nitrobenzylideneamino)-N-(pyrimidin-2-yl)-3-(thiazol-2-yl diazenyl) benzene sulfonamid]., and (Z4) [4-((Z)-4-chlorobenzylideneamino)-N-(pyrimidin-2-yl)-3-(thiazol-2-yldia zenyl) benzenesulfonamide] :

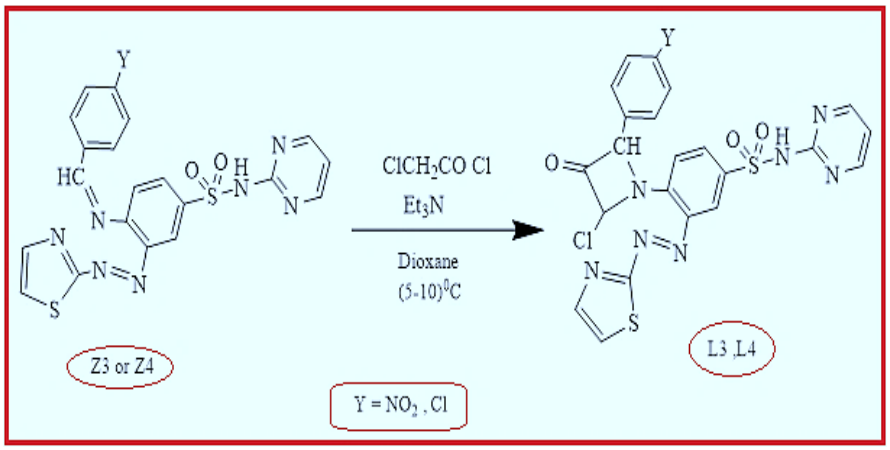
Azo Compound(Z1) (0.001 mol,0.36 gm) mixed with aldehydes in acidic medium,, by using (Benz: MeOH, 2: 3) as mobile phase to format product. (Z3) ($C_{20}H_{14}N_8O_4S_2$) yield: Dark orange 55%, m.p.(134-136) ^{0}C, R_f (0.79) ., (Z4)($C_{20}H_{14}ClN_7O_2S_2$) yield: Maroon 50%, m.p.(222-224) ^{0}C, R_f (0.84).

Overall practice for derivatives(L3, L4).

To a mixture of Z3, Z4 (0.001 mol,0.49gm),(0.001 mol,0.48gm)respectively in dioxane (25 ml) and Tri ethylamine (0.35 ml,0.0025 mol), halo alkoyl halide(0.2 ml, 0.0025 mol) was further slowly for two days to give product. (L3): ($C_{22}H_{15}ClN_8O_5S_2$) yield: Brown ,45%, m.p.(Oily), R_f (0.54). (L4): ($C_{22}H_{15}Cl_2N_7O_3S_2$) yield: Dark Brown ,45%, m.p.(Decomp. 300) ^{0}C, R_f (0. 47).



Equation.1: Preparation of Azo –Pyrimidine comps. (Z1, Z2, Z3, Z4)



Equation.2: Preparation of Azo –Pyrimidine comps. (L1, L2)

3. RESULTS AND DICUSSION:

Steps of reaction with P-Amino acetophenone and sulfadiazine respectively above in **Equations(1** , 2)., The Azo compounds were identified by bands of stretching band of a primary aromatic amine (NH_2) in Z1 (one band overlapping with NH Sulfone amide in 3379-3217) and Azo group (N=N) at (1438-1413),(1436-1409) cm^{-1}, (C=N)endo cyclic (1598,1637) cm^{-1}, respectively. At the same time, there were identified in A1, a stretching band of ketone carbonyl at(1672) cm^{-1},andstretching band of SO_2 at(1257) cm^{-1}in Z1., 1HNMR: (S, 3H,CH_3-),(S,2H,NH_2 ,) 7.41,(m,2Hfor thiazole ring) (8.1-8.63) while carbon resonance gave Azo group N=N)155.43, C (Aromatic rings)121.90-

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152.04,C(-C=O)197.05, DMSO (solvent) 40.2., also (Z1) gave (NH Sulfon amide)11.43,(S,2H,NH_2 ,) 6.03,(m,5H for thiazole and pyrimidine rings) (8.48-8.53) ,and(m,3H for benzene ring) (6.55-7.64). (Thiazole ring Associated with Azo group N=N)158.71, and C (Aromatic rings)112.57-157.67., 1H-NMR spectrum (ppm)(DMSO-*d6*) for(Z2) , showed characteristic signals at ppm: (S,1H.NH,Amide) at (8.55),(S,1H,NH,sulfon amide) (11.4),(m ,2Hfor thiazole ring) (8.50-8. 53) ,(m,6H for aromatic rings) (6.55-8.4),and (S,2H.CH_2-C=O)(4.23). The signal (3.49) ppmwas due to the solvent DMSO-*d6* and water dissolved in DMSO,(2.5)ppmwasdue to the solvent DMSO-*d6*., The derivatives (Z3,Z4) were prepared from Azo comp. (Z1) by condensation reaction with P-Nitro benzaldehyde and P-Chloro benzaldehyde respectively by using hot glacial acetic acid as solvent. The new derivatives were identified by FT-IR spectra, figures (20 and 21). (Z3)was identified through the disappearance of the stretching bands for aromatic primary amine and appearance of a stretching band at 3381 cm^{-1} due to (NH)sulfon amide, stretching bands, one at1521cm^{-1}due to Schiff base(C=N) and another at 1581 cm^{1}due to (C=N) endo cyclic, Azo group (N=N) at (1440-1409) cm^{-1}, (C=C) Aromatic at (1492)cm^{-1}, and a stretching band of SO_2 at(1255)cm^{-1}, in addition to stretching band of(C-S) at (945) cm^{-1},andfinally (C-H) aromatic at (3084-3039)cm^{-1}. (Z4)was characterized by the appearance of a stretching band at 3421 cm^{-1} due to (NH)sulfon amide overlapping with (C-H) aromatic, and the appearance of a stretching band at1581cm^{-1}due to Schiff base(C=N) but at 1637 cm^{-1}due to (C=N) endo cyclic, Azo group (N=N) at (1417) cm^{-1}, (C=C) Aromatic at (1550)cm^{-1} and a stretching band of SO_2 at(1238)cm^{-1}, and finally stretching band of(C-S) at (945) cm^{-1}., 1H-NMR spectrum (ppm)(DMSO-*d6*) for(Z3) , showed characteristic signals at ppm:(1H,CH=N) **[24]** imine group at (9.5),(S,1H.NH,Sulfon Amide at (11.27),(m ,2Hfor thiazole ring) (8.48-8. 51),(m,7H for aromatic rings) (6.0-8.0). , While figure 26, showed the typical resonance of carbonyl group "β-Lactam" derivative at (169.48). (C)CH-Cl ,58.229, (C) CH-N ,64.623 , (C)phenyl rings, 118.611-143.537, (C) Pyrimidine ring ,157. 656, 116.023,(C-N=N)158.771 .

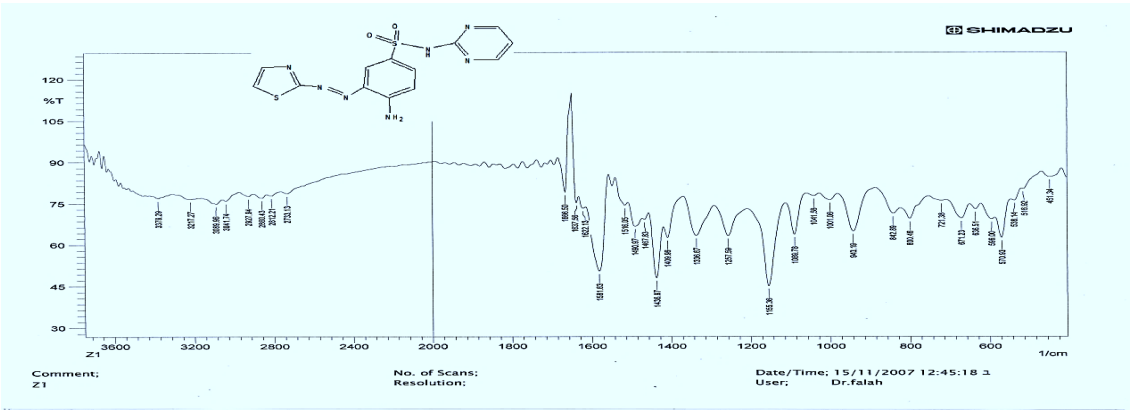

Figure (1) FT-IR spectrum for Z1

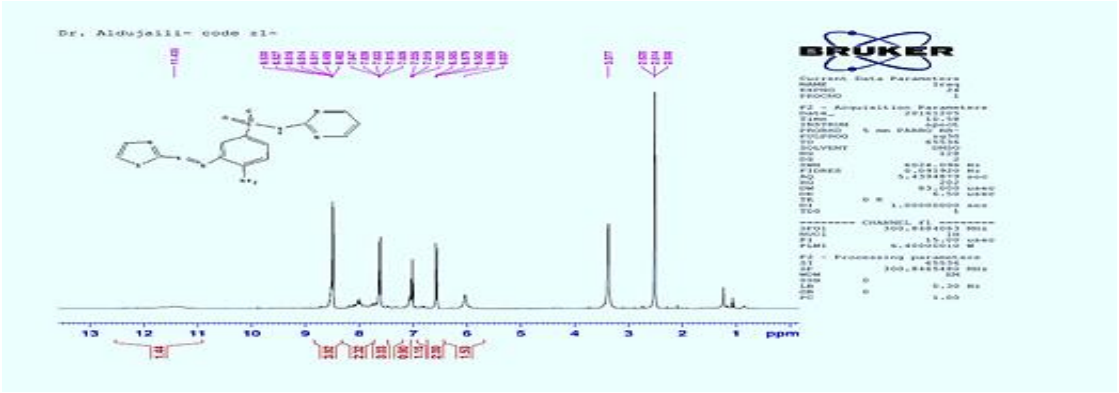

Figure (2) 1H-NMR spectrum for Z1

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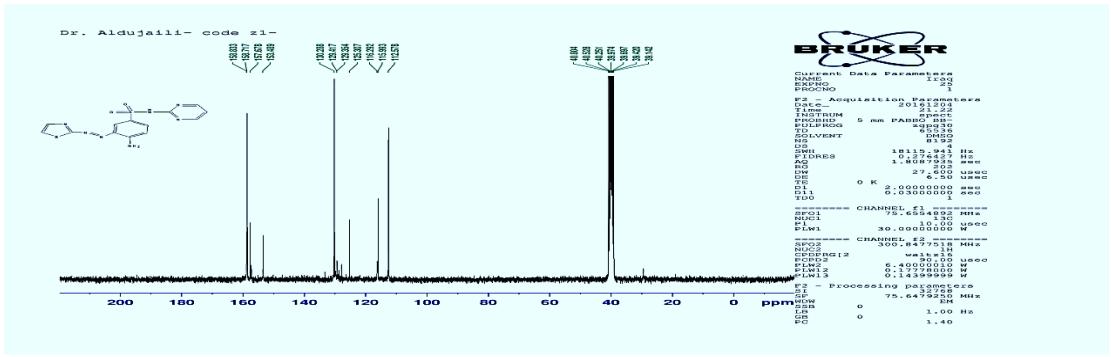

Figure (3) 13C-NMR spectrum for Z1

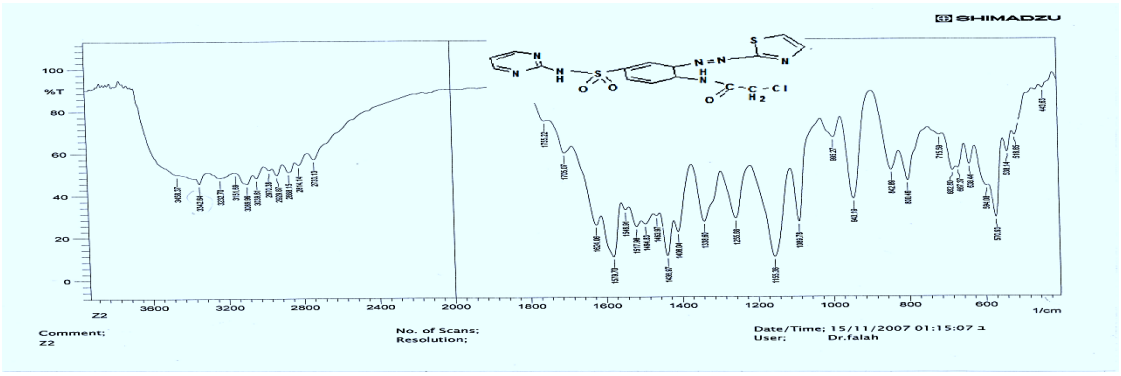

Figure (4) FT-IR spectrum for Z2

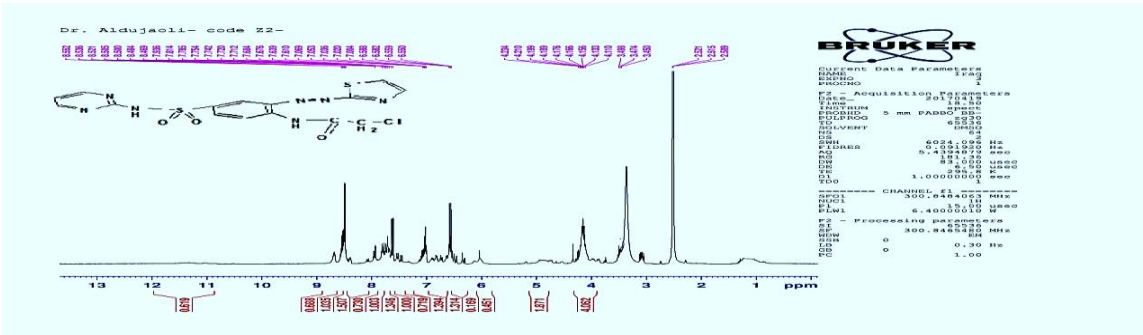

Figure (5) 1H-NMR spectrum for Z2

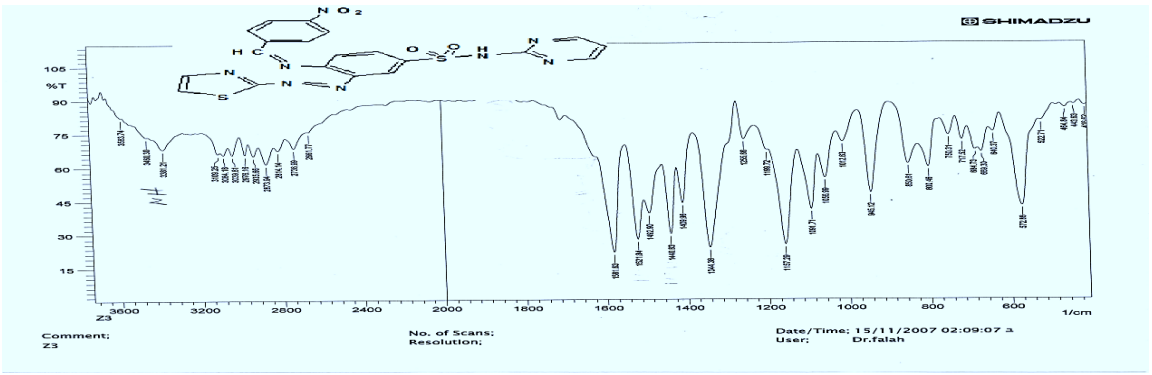

Figure (6) FT-IR spectrum for Z3

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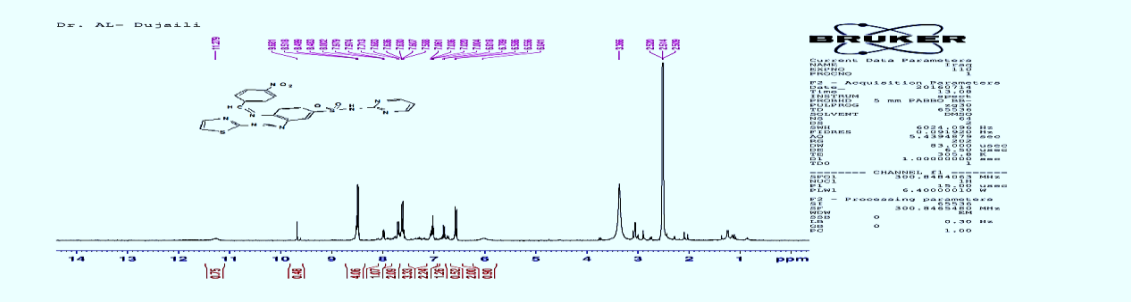

Figure (7) 1H-NMR spectrum for Z3

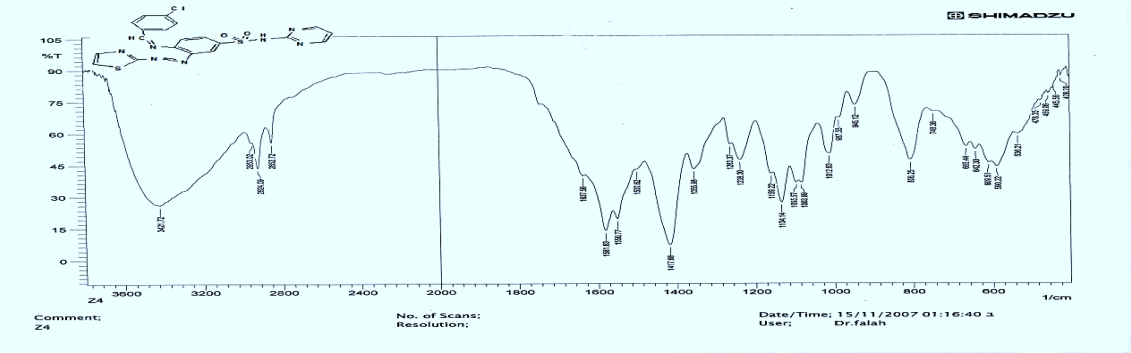

Figure (8) FT-IR spectrum for Z4

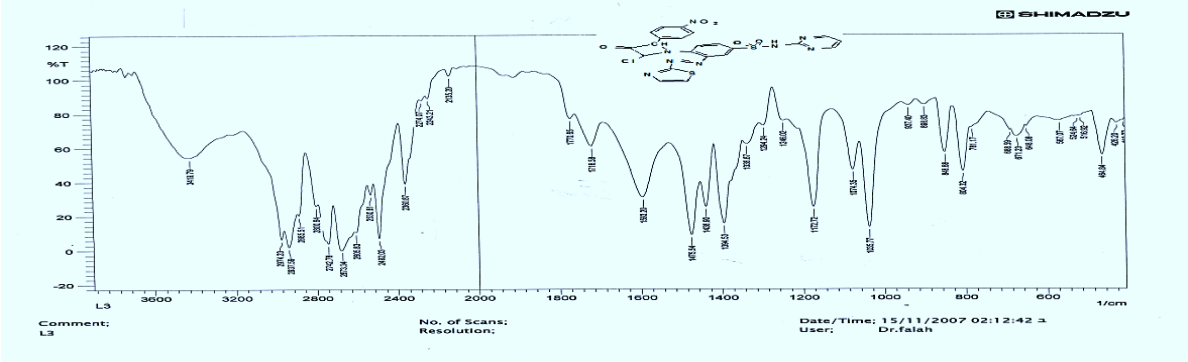

Figure (9) FT-IR spectrum for L3

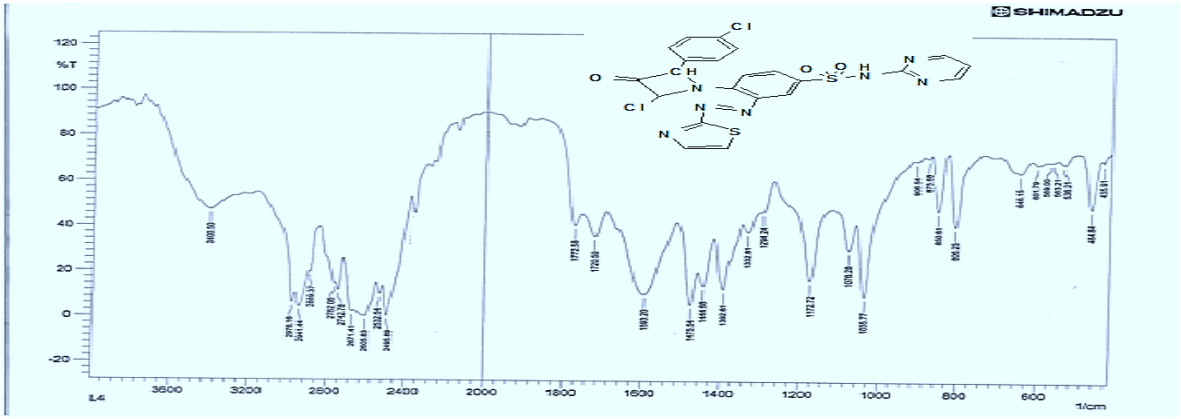

Figure (10) FT-IR spectrum for L4

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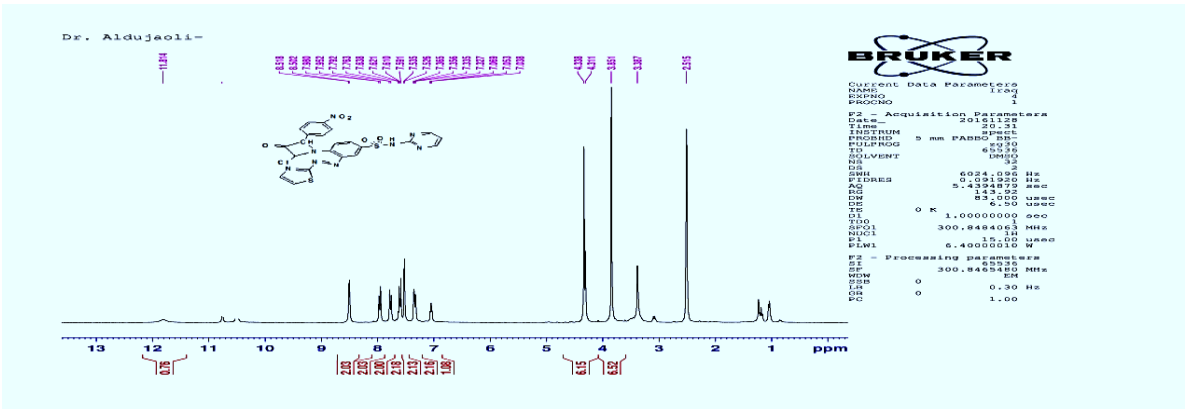

Figure (11) 1H-NMR spectrum for L3

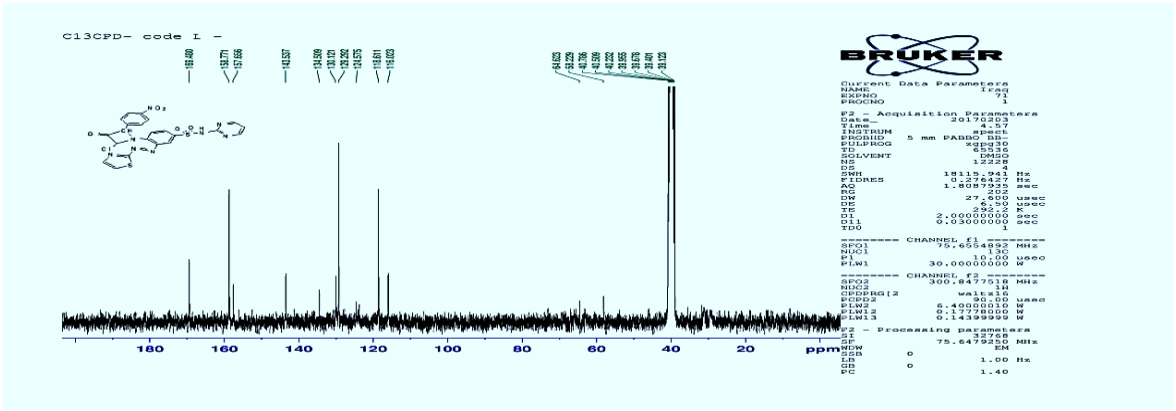

Figure (12) 13C-NMR spectrum for L3

Table1: The physical belongings

Comp.	MF	MWt (g/mol)	MP (c°)	Color	Vintage %	R_f
Z_1	$C_{13}H_{11}N_7O_2S_2$	361	(184-186)	Dark orange	50	
Z_2	$C_{15}H_{12}ClN_7O_3S_2$	437.5	(177-179)	Light orange	53	0.88 (Benz: MeOH (2: 3)
Z_3	$C_{20}H_{14}N_8O_4S_2$	494	(134-136)	Dark orange	55	0.79 (Benz: MeOH) (2: 3)
Z_4	$C_{20}H_{14}ClN_7O_2S_2$	483.5	(222-224)	Maroon	50%	0.84 (Benz: MeOH) (2: 3)
L_3	$C_{22}H_{15}ClN_8O_5S_2$	570.5	Oily	Brown	45	0.54 (Benz: MeOH) (2: 3)

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L_4	$C_{22}H_{15}Cl_2N_7O_3S_2$	560	Dicomp. 300	Dark brown	45	0.47 (Benz: MeOH) (2: 3)

Anti-fungal Activity

The biological activity of some prepared derivatives(Z1,Z2,Z3,L3,L4)was examined in this work against type of Fungi, namely*(Fusarimoxy sporium)* , by using potatose dextrose Agar(PDA)as culture medium. The used concentration for examined derivatives was(0.0001 M) in DMSO as solvent [43-46]. The examined derivatives exhibited moderate activity as in table(2).

Table (2): Inhibition Zones

Comp.(0.0001M)	Inhibition Zone/ mm
Z1	3
Z2	3
Z3	7
L3	3
L4	3

ASSUMPTION

The recent work indicated that derivatives is slick,. In addition, the biological evaluation confirmed that some of them are anti-fungal and gave good results towards fungi by inhibition of wall of cells.

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Nil

AUTHORS CONTRIBUTIONS

The author carried out and contributed in all experiments ,writing, analysis ,identification .

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