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ت	فهرس البحوث	الصفحة
1	Estimate of cold plasma on antibiotic resistance and biofilm formation in Staphylococcus aureus isolated from clinical cases Zainab Sabah Fahim Majid Kadhim Aboud Al Shibly	1 - 13
2	The Subjugation of Bodies, Gender, and Biopolitics in Nawal El Saadawi's Woman at Point Zero Ali Mohammed Hasan	14 - 23
3	A Comparative study between Boundary and Finite Element Techniques for solving Inverse Problems Farah A.Saeed Sarah F. Ghafel	24 - 33
4	Postcolonial Feminism and Political Injustice in Huda Barakat's The Tiller of Waters Afrah Abdul Jabbar AbdulSahib	34 - 47
5	Cordia Myxa Fruit Effect on Bacterial Adhesion to Heat-Cured Acrylic Denture Bases Noor R. Taha Shorouq M. Abass	48 - 60
6	Prevalence and detection of Yersinia enterocolitica isolated from different clinical cases Baneen Maan Kareem Hadaf Mahdi Kadhim	61 - 74
7	Molecular investigation of biofilm genes in Staphylococcus epidermidis Lamyaa Jabbar Abosaooda Baheeja Abees Hmood Al-Khalidi	75 - 81
8	Salivary Thiocyanate Levels and Buccal Mucosal Cells Changes in E-cigarette Users and Traditional Smokers Mufeed Muhammad Jawad Yas, Layla Sabri	82 - 101
9	The Metaphorical Representation of Coronavirus in Iraqi Newspaper Cartoons Hayder Tuama Jasim Al-Saedi	102 - 142
10	The Narrative Synthesis of Human Frailty and the Social Attributes of the Singaporean Society in Philip Jeyaretnam's Abraham's Promise" Rana Ali Mhoodar	143 - 151
11	Development of Thiadiazole and Schiff Base Derivatives: Synthesis, Spectral Characterization, and Antibacterial Activity Assessment Doha kareem Hussien Rafid Kais Kmal Haitham Kadhim Dakheel	152 - 161
12	Translating voices from the Tigris: The American Granddaughter as a case study Falah Hussein Hanoon Al-Sari	162-172
13	Synthesis and Characterization of Conductive Copolymer/MWCNT Nanocomposite via Chemical and Interfacial Polymerization Hajer A. Hussein Mohammed Q Mohammed	173 - 186
14	Synthesis, diagnosis, and study of the electrical properties of some new iron-polymer complexes containing Schiff bases and study of their thermal stability Abduljaleel Muhammad Abduljaleel Nadia Ashour Hussein Jassim Mohammed Saleh	187 - 205

206 - 221	Reptiton in Surah Ghafir, Fussilt, and Ash-shura An - Applied Study Qusay Tawfiq Hantoush	**15**
222 - 233	The short story in the literature of Said Hashosh, with 'The White Rainbow' as a model. Rabab Hussain Muneer	**16**
234 - 249	Aesthetic functions in men's fabric and fashion designs Asaad Ati Halil Saad Al-Moussawi	**17**
250 - 264	Techniques and Methods of Deviation in the Poetry of Kazem Al-Hajjaj and Mohammed Al-Khafaji Imad Hameed Nassrah Al-Musaedi Danesh Mohammadi Rakati Yusuf Nazari	**18**
265 - 279	Assimilation and Its Impact on Morphological Structure A Phonological and Morphological Study of Poetic Samples from Various Historical Periods Suad Abbas Sayyid	**19**
280 - 302	The Degree to Which Middle School Mathematics Teachers Possess Creative Teaching Skills Saif Karim Muslim	**20**
303 - 317	Plantinga's Epistemological Justification for General Beliefs Sajjad Saleh ShenyarAbbas Mahdavi Mostafa Farhoudi Mohammad Keyvanfar	**21**
318 - 333	The effect of the situation on leaving out the forgotten non-metaphorical object in the Holy Qur'an Yassin Taher Ayez	**22**
334 - 349	Ambiguities and Their Role in Constructing Stylistic Connotation: A Reading in Ahmed Zeki Abo Shadi's Alyanbu'a Poetry Rania Ali Munim	**23**
350 - 368	The reality of Primary School Teachers' Practice of Social Sciences to The Contemporary Professional Criteria Qasim Jaleel Zayir Al-Ghurabi Ramla Jabbar Khadhm Al-Saedi	**24**
369 - 383	Rhythm techniques in the poetry of Hasab Al-Sheikh Jaafar Mayyada AbduLAmeer ISmael	**25**
384 - 393	Youm AlHashir in Holy Quran: Surat Alansan as A Case Study Ali Howair Swailem	**26**
394 - 408	Criminal Policy for Addressing Crimes of Disclosure of Occupational Secrets and Violations of Messages and Telegrams (Comparative Study) Mustafa Shakir Hussein	**27**

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Development of Thiadiazole and Schiff Base Derivatives: Synthesis, Spectral Characterization, and Antibacterial Activity Assessment

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Abstract:

Given the significant importance and distinctive applications of Schiff base compounds in both academic and applied fields, this study focused on the synthesis of a new heterocyclic compound. Several spectroscopic techniques were employed to characterize the synthesized compound after precipitation and purification. These techniques included: Proton nuclear magnetic resonance spectroscopy (^{1}H-NMR) and Fourier-transform infrared spectroscopy (FT-IR). The solubility of the compound was also evaluated. It was found to be highly soluble in ethanol, methanol, dimethyl sulfoxide (DMSO), and tetrahydrofuran (THF), whereas it was insoluble in water and diethyl ether , In addition, the biological activity of the synthesized compound was investigated against selected strains of Gram-positive and Gram-negative bacteria. The compound exhibited moderate to high antibacterial activity, which suggests its potential as a candidate for further pharmacological development.

Keywords: heterogeneous , (FT-IR) , spectroscopy, synthesis, biological activity

1. Introduction:

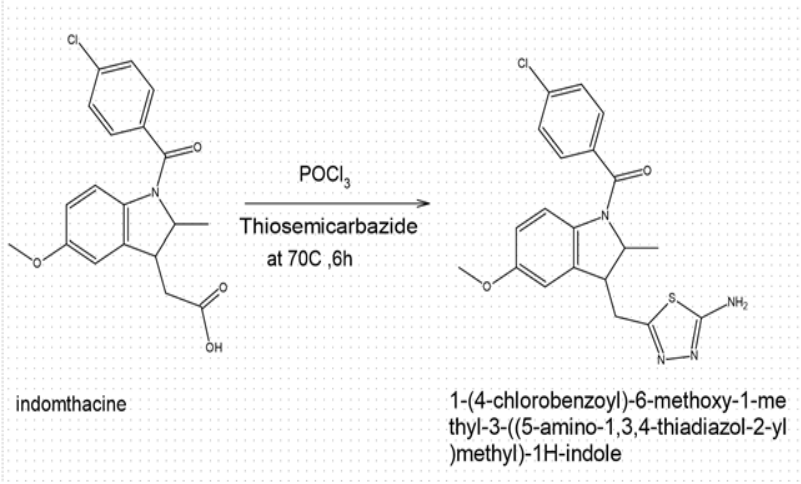
Heterocyclic compounds that contain both the azo bridge group (-N=N-) and the azomethine group (C=N-) represent an important class of organic compounds due to their high reactivity toward most elements of the periodic table. This is attributed to the presence of multiple functional groups capable of forming chelate complexes with metal ions. These metal complexes are characterized by their color and their solubility in both polar and non-polar solvents (Karmakar. 2020), (Sumrra. 2021) . Azomethine compounds have attracted significant attention in numerous studies because of their wide range of applications, particularly in organic synthesis and biological activities, especially in the medical field. In addition, these compounds exhibit a high ability to coordinate with various metal ions, resulting in the formation of stable complexes. This coordination ability arises from the presence of two key functional groups in their chemical

structure the azomethine (-C=N-) group, which impart unique properties to these compounds(Al-Khateeb. 2019), (Tarhriz. 2020), (Marinova. 2011) .

2.Experimental:

2.1. Preparation of 1-(4-chlorobenzoyl)-6-methoxy-1-methyl-3-((5-amino-1,3,4-thiadiazol-2-yl)methyl)-1H-indole

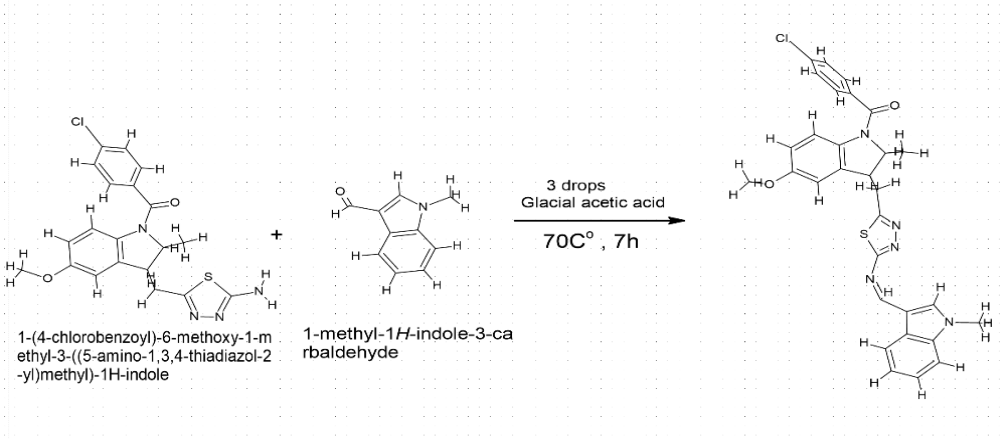
A mixture of Indomethacin (0.001 mol, 0.35779 g) and thiosemicarbazide (0.001 mol, 0.09114 g) was placed in a two-neck round-bottom flask equipped with a magnetic stirrer. The mixture was dissolved by gradually adding 15 mL of phosphorus oxychloride ($POCl_3$), then refluxed for three hours at 80°C. The resulting solution was cooled to room temperature, and 40 mL of distilled water was slowly added dropwise. The mixture was then refluxed again for four hours. The progress of the reaction was monitored using TLC (benzene:methanol = 4:1). After completion, the reaction mixture was cooled to 25°C, filtered, and the filtrate was neutralized using an aqueous solution of 50% potassium hydroxide. A yellow precipitate formed, which was collected by filtration, washed with distilled water several times, dried, and recrystallized. The final product was weighed, and its percentage yield and physical properties were recorded(Alabidi. 2021) ,(Dakheel. 2022) .



Scheme 1. Synthetic of of new 1-(4-chlorobenzoyl)-6-methoxy-1-methyl-3-((5-amino-1,3,4-thiadiazol-2-yl)methyl)-1H-indole

2.2. Synthesis of Schiff Base (L_1)

The compound (L1) was synthesized by dissolving 0.36 g (0.0041 mol) of the previously prepared thiadiazole compound with 0.14 g (0.00088 mol) of 1-methyl-1H-indole-3-carbaldehyde in 25 mL of absolute ethanol. To this solution, 2–3 drops of glacial acetic acid were added. The resulting mixture was refluxed for 2 hours, then cooled, leading to the formation of a precipitate. The reaction mixture was left to stand for 24 hours, after which the precipitate was filtered and dried. The product was identified, recrystallized from absolute ethanol, and collected to give a yield of 85% with a recorded melting point(Salmam. 2021), (Asharani. 2009) .



Scheme2. Synthetic of of new Schiff Base (L1)

3.Discussion:

3.1. H[1]-NMR spectra:

The ¹H-NMR spectrum of thiadiazole compound (1) was recorded in DMSO-d_6 as solvent, using TMS as an internal standard at room temperature, and compared with literature data. The spectrum showed a singlet at δ = 1.77 ppm, attributed to the CH_3 protons; a signal at δ = 3.75 ppm corresponding to the methoxy group; and a signal at δ = 5.18 ppm attributed to the NH_2 group protons. A multiplet was observed in the range δ = 6.52–8.59 ppm, corresponding to the aromatic protons.Additionally, a singlet at δ = 2.18 ppm was assigned to CH_2 protons, and a signal at δ = 3.76 ppm was attributed to the indole ring protons. A peak at δ = 2.52 ppm corresponds to the DMSO-d_6 solvent. Table (3) presents the chemical shift values of ¹H-NMR signals for thiadiazole compound (1). The ¹H-NMR spectrum of the Schiff base compound (2) also showed characteristic signals. A singlet at δ = 1.89 ppm was assigned to CH_3 protons, and another at δ = 3.89 ppm for methoxy group protons. A signal at δ = 2.99 ppm corresponds to the N-CH_3 protons, while a multiplet in the region δ = 7.07–8.54 ppm was attributed to aromatic protons.

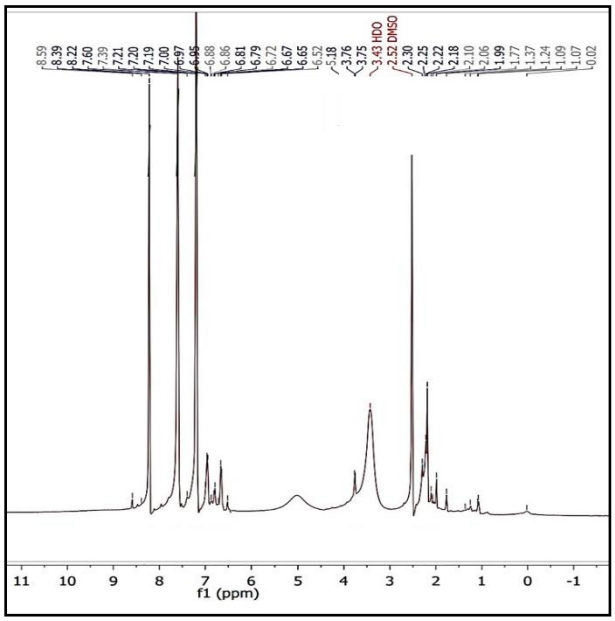
Additionally, a singlet at δ = 2.33 ppm corresponds to CH_2 protons, and a signal at δ = 3.42 ppm was assigned to the indole ring protons(Al-Adilee.2020) . The solvent DMSO-d_6 also appeared at δ = 2.52 ppm.Table (4) shows the ¹H-NMR spectral data for Schiff base compound (2).

Table (1): ¹H-NMR Chemical Shift Values for the Thiadiazole Compound (2)

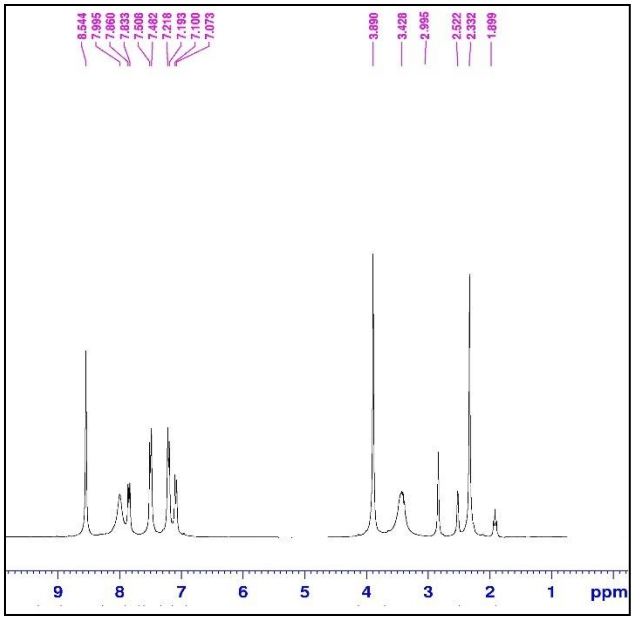
Associated group	Signals (ppm)
-CH^3	(δ=1.77)
-O-CH_3	(δ=3.75)
CH_2-	(δ=2.18)
NH^2 -	(δ=5.18)
Phenyl ring	(δ=6.52 - 8.59)
CH indole-	(δ=3.76)
Solvent (DMSO-d_6)	(δ=2.52)

Table (2): ^{1}H-NMR Chemical Shift Values for the Schiff Base Compound (2)

Associated group	Signals (ppm)
-CH^{3}	(δ=1.899)
-O-CH_3	(δ=3.89)
CH_2-	(δ=2.33)
N-CH_3	(δ=2.99)
Phenyl ring	(δ=7.07 - 8.54)
CH indole-	(δ=3.42)
Solvent (DMSO-d_6)	(δ=2.52)



Figur 1 proton magnetic resonance Thiadiazole in DMSO solven



Figur 2- proton magnetic resonance spectrum of spectrum of Schiff Base in DMSO solvent

The thiadiazole compound (1) was characterized using Fourier-Transform Infrared (FT-IR) spectroscopy, where the disappearance of certain absorption bands and the appearance of a new band at 3551 cm^{-1}, corresponding to the N–H stretching vibration, were observed, as shown in Figure (3). Table (1) presents the remaining absorption band values for thiadiazole compound (1).

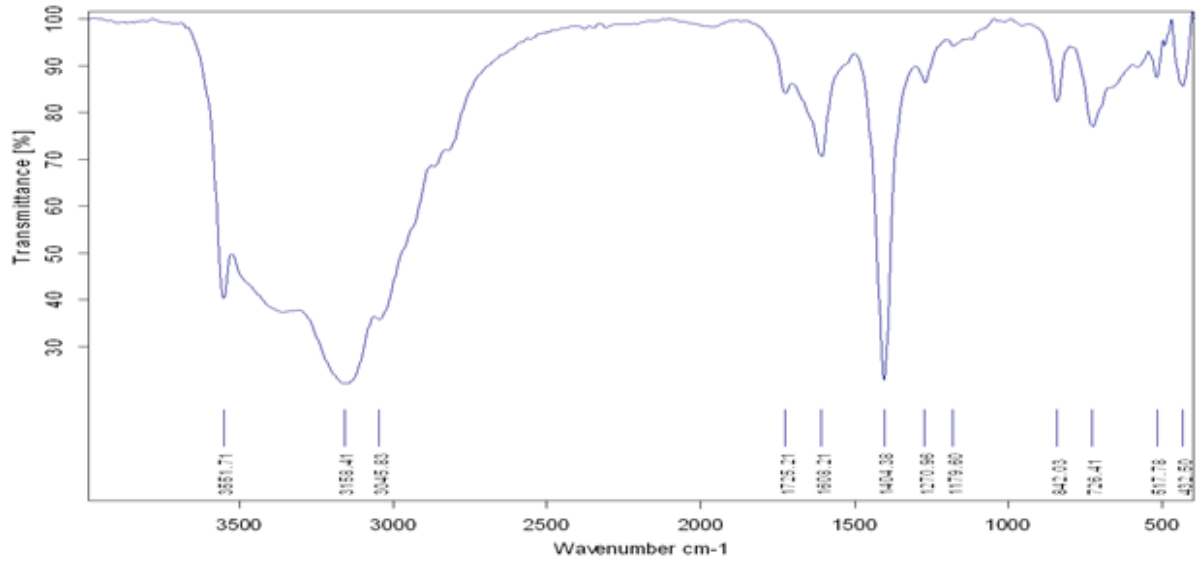
Similarly, the Schiff base compound (2) was characterized using FT-IR spectroscopy, where the disappearance of the band at 3551 cm^{-1}, corresponding to the N–H stretching vibration, indicates the formation of the azomethine (C=N) bond. This is also shown in Figure (4), and Table (2) displays the rest of the FT-IR absorption bands for the compound under study(Dakheel .2023) .

Table (3): FT-IR Absorption Band Values for Thiadiazole Compound (1)

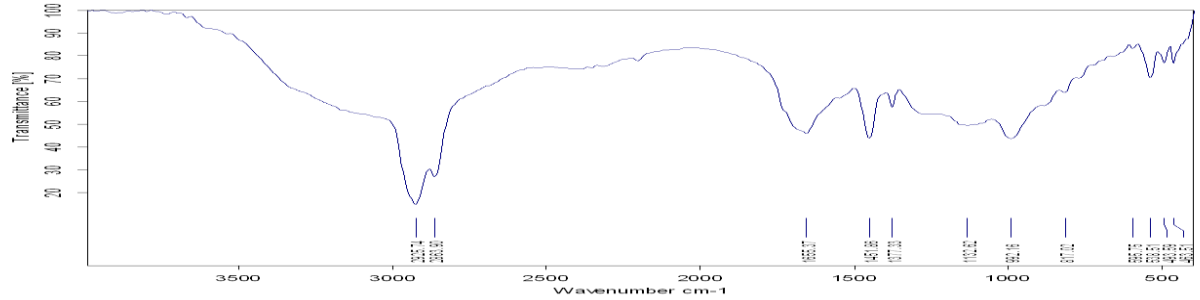
Comp.	υ(C-H) arom. St.	υ(C-H) aliph. St.	υ(N-H) St.	υ(C=N) St.	υ(C=C) St.	υ(C=O) St.	υ(C-N) St.	υ(C-S) St.
1	3158	3045	3551	1608	1404	1726	1270	1179

Table (4): FT-IR Absorption Band Values for Schiff base Compound (1)

Comp.	υ(C-H) arom. St.	υ(C-H) aliph. St.	υ(N-H) St.	υ(C=N) St.	υ(C=C) St.	υ(C=O) St.	υ(C-N) St.	υ(C-S) St.
1	2925	2863	------	1451	1377	1655	1231	1132



Figur (3): FT-IR Absorption Band Values for Thiadiazole Compound (1)



Figur (4): FT-IR Absorption Band Values for Schiff base Compound (1)

Mass spectrometric analysis of the thiadiazole compound revealed several peaks, the most significant of which appeared at m/z = 413.9, corresponding to the molecular ion peak of the synthesized thiadiazole compound with the formula $C_{20}H_{19}ClN_4O_2S$.

The mass spectrum of the Schiff base compound (2) also exhibited multiple peaks, with a major peak at m/z = 556, corresponding to the molecular ion of the synthesized compound with the molecular formula $C_{30}H_{26}ClN_5O_2S$. The figures below illustrate some of the spectral measurements obtained for the two synthesized compounds(Venugopal.2020) .

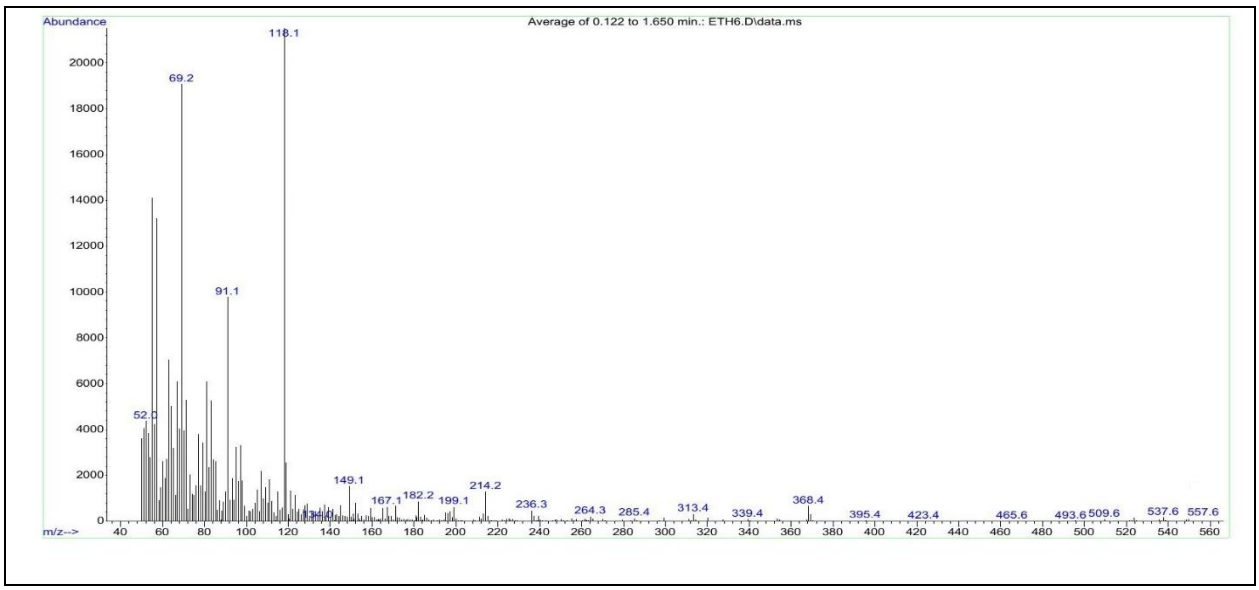


Figure (5): Mass Spectrum of the Thiadiazole Compound

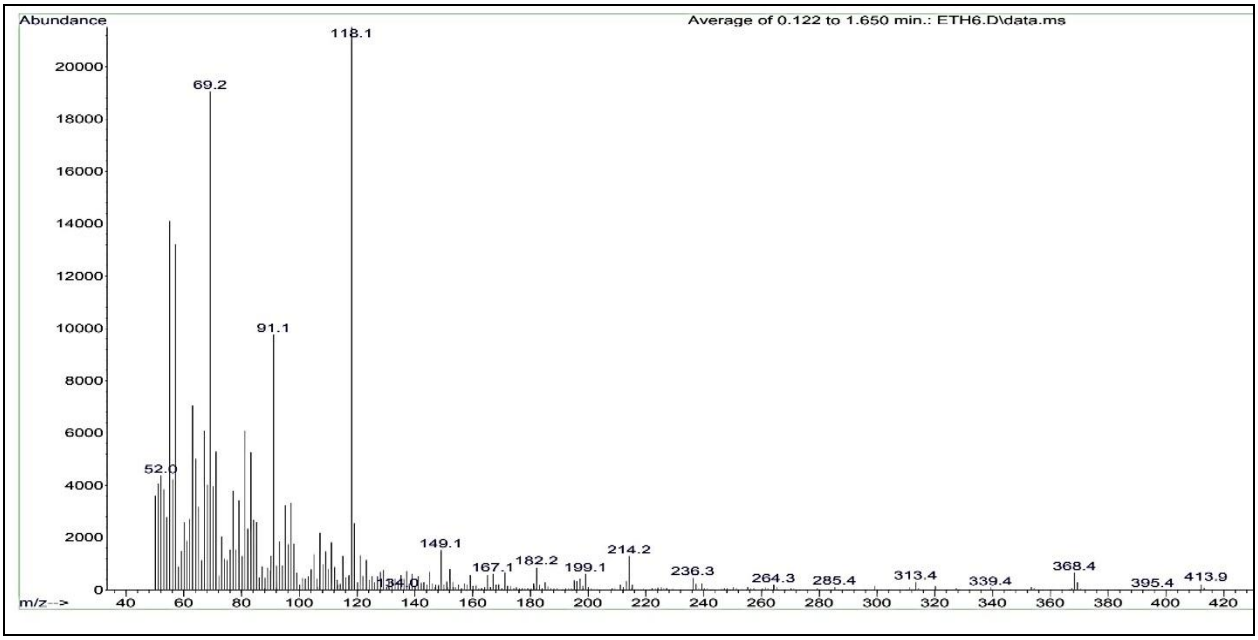


Figure (6): Mass Spectrum of the Schiff base Compound

4.Biological activity:

Heterocyclic compounds represent an important class of organic compounds, as their compounds possess distinct biological and pharmacological properties. Due to their remarkable biological properties, these heterocyclic derivatives, either alone or in combination with other groups, have been exploited by researchers to develop new drugs (Nath.2020) . In the past 100 years, some new antibiotics have been developed, but bacterial resistance remains a major global problem. There are few solutions being explored, but the misuse of antibiotics has led to the emergence of resistant strains, which increases the risk of bacterial infections in humans. Bacterial diseases have caused many deaths worldwide in the past (Kumar . 2020) . Fortunately, since the discovery of penicillin in the 1940s (Chen. 2023), (Guo. 2023) , (Rusu.2023) many natural and synthetic antibiotics have significantly improved human health. However, the challenge lies in treating bacterial infections due to the emergence of drug-resistant strains and the increasing prevalence of infectious diseases. Despite the availability of various antibacterial agents, the emergence of resistant bacterial strains in recent years highlights the urgent need for new classes of antibiotics (Kanagavalli. 2023), (Abu-Yamin.2022), (Naureen. 2021) . Heterocyclic antibiotics are a class of antimicrobial agents that possess a ring structure containing atoms of at least two different elements, usually carbon and nitrogen, within the ring. These heterocyclic compounds exhibit powerful antimicrobial properties and have been essential in the treatment of various bacterial infections (Reddy.2023) , (Jorge. 2024) , (Rana. 2025) . In our study, we will address two types of Gram-negative and Gram-positive bacteria: Staphylococcus aureus and Escherichia coli. It was observed that thiadiazole was more inhibitory to bacteria than the compound 1H-1-methyl-indole-3-carboxydehyde through the measurements shown in the table above

Table (5): Antibacterial Activity (Inhibition Zone in mm) of the Synthesized Compounds Against *Escherichia coli* and *Staphylococcus aureus*

Name of compound	Staphylococcus Aureus mm	Escherichia Coli Mm
1-(4-chlorobenzoyl)-6-methoxy-1-methyl-3-((5-amino-1,3,4-thiadiazol-2-yl)methyl)-1H-indole	1	1.3
Schiff Base (L_1)	0.6	0.5

5. Conclusions:

Based on the diagnostic and spectroscopic results of the two compounds under study, it is recommended to investigate the biological activity of the synthesized

compounds. In addition, due to their intense and vivid coloration, these compounds may also find potential applications in the field of analytical chemistry.

Declaration of Competing Interest:

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Conflicts of Interest Statement……

Manuscript title: **Development of Thiadiazole and Schiff Base Derivatives: Synthesis, Spectral Characterization, and Antibacterial Activity Assessment**

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