

Synthesis and Properties of Tetrazole Compounds on Adamantan's Schiff Bases Derivatives

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ABSTRACT

This research includes the preparation of azo-schiff compounds derived from amino salicylic acid, and that is through linking with the first amine group after converting it to diazonium salt and its interaction with aromatic compounds containing a group (aldehyde or ketone) and the formation of azo and through the carbonyl group of the aldehyde or ketone, it reacts again with compounds containing the first amine group to form Schiff bases. Through the imine bond, we work to form heterogeneous rings to obtain compounds with industrial or medical benefits. Preparation of azo compounds (H1-H8) through the transformation of 5-amino salicylic acid into diazonium salt to be reacted later with a number of aromatic compounds (aldehydes or ketones). The compounds prepared were diagnosed using FT-IR and H-NMR technology for some of the prepared compounds and their physical properties. Studying the biological activity of some of the prepared compounds.

KEYWORDS: Heterocyclic, azo compounds, Diazonium, Azide, Schiff base.

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INTRODUCTION

The mesalazine compound, known as mesalamine [1] and the figure 1 represents the formula for the formulation of 5-amino salicylic acid.

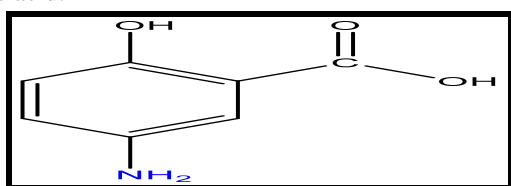


Figure 1: structure of 5-aminosalicylic acids compound

Amino salicylic acid (5-ASA) is the most widely prescribed anti-inflammatory drug for inflammatory bowel disease IBD[2], and is also used to treat ulcerative colitis in the gastrointestinal tract [3]. The 5-ASA was approved by the Food and Drug Administration (FDA) in 1987 [4], and in the market is known for various brands such as mesalamine, mesalazine and ask.

AZO COMPOUNDS

Are compounds containing one or more groups of nitrogen atoms in which SP²[5]. In 1858 (Grisis Peter) found that aromatic amines react quickly with nitrous acid at low temperatures to form easily soluble salts in water known as

diazonium salt (diazonium salt) [6]. In (1860) the Azo(-N=N-) [7] a hybrid was able (Grisis) to detect this type of organic compound and called Azo compounds as a person having two nitrogen atoms bonded together by a contemporary Double ((-N=N- in molecule[8]), The stability of aromatic azo compound is due to the fact that they contain the strong double-bonded azo group and the stability of this type of compound is affected by the type of aggregates (-N=N-) associated on both sides of the azo group and the number of such aggregates. When aggregates contain double bonds in sequence with the double azo group and other aromatic groups, the compound becomes highly stable due to the occurrence of the phenomenon of the resonance between the double corner of the Azo group and the other double bonds of the groups as shown in the figure 2. in the azo-benzene compound [9], and because of its physical and chemical properties in addition to its biological effectiveness. It has many important applications in pharmaceuticals, cosmetics, textile industry, analytical chemistry, and food and is known for its medicinal significance, and is recognized in many applications such as antidiabetics[5]. It is involved in many biological reactions such as RNA's RNA's DNA inhibition, DNADNA, protein synthesis, and nitrogen fixation[10]. These compounds possess biological effectiveness as

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antibacterial [11], antitumor [12] and active insecticides [13, 14]

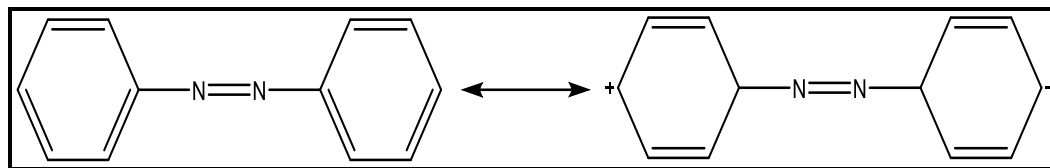
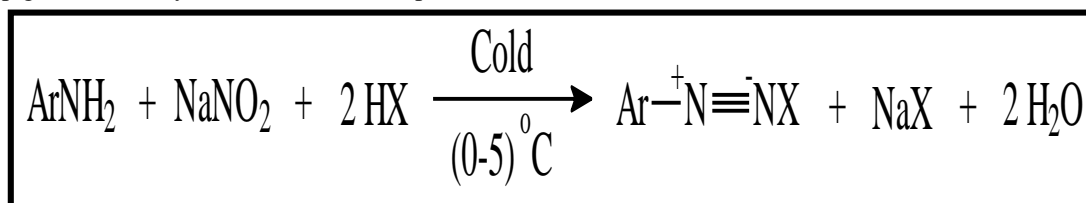


Figure 2: Azo benzene compound

Azo compounds are important for the preparation of a large number of compounds and pure organic substances [15]. As a result of possessing electrolytic properties, diazonium salts [16] have the ability to pair with many high-density electronic compounds. Diazonium salts also play an active role in the chemistry of pigments as they are associated with phenols,

naphtha's and ethyl amine, giving many types of color. Diazonium salts [17,18] consist of the initial aromatic amine treatment with nitrite ion in an acidic. At the degree (0-5) this reaction is called as a mate as shown in equation below;



x= Cl, Br, NO₃, HSO₄

Diazonium salt

The factors affecting the stability of diazonium salt are the composition of the salt and its concentration in the solution as well as the nature of the compensating aggregates on the amine. Tug aggregates reduce the stability of diazonium salt, while propellant aggregates increase its stability [19]. The

interaction of nitrous acid with primary aromatic amines is an important reaction widely used in organic chemistry, giving aromatic diazonium salts that are relatively stable [20]. Diazonium salts are one of the most important compounds used in the preparation of many organic compounds.

SCHIFF BASES

It is a class of organic compounds that in their chemical composition contain the azomethine group (C=N) as an effective group [21]. Schiff's rules were obtained by an interplay of condensation between the first two secretaries with the carbonyl group and she was first attended by German scientist Hugo Schiff Hugo Schiff in 1864[22]. In terms of composition. The Schiff base (also known as right or azomethane) is an aldehyde or ketone nitrogen isotope where it contains the carbonyl group (C=O) replaced by the imine or isomethane [23] as shown in the figure3 below:

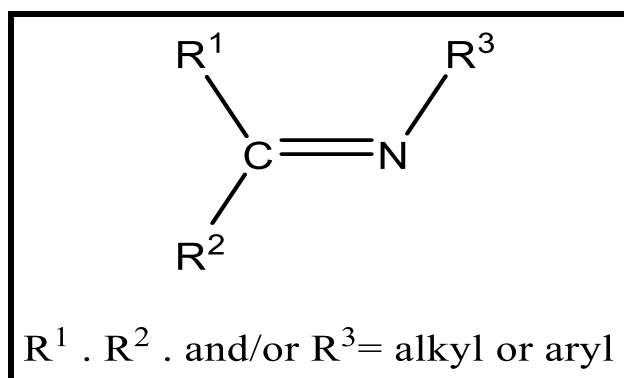


Figure 3: Azsomethane

Schiff's rules have gained importance due to their use in many pharmacological activities such as antibacterial [24,25], antifungal [26], anti-reproduction[27], and anti-tumor[28]. The rules of XF have been widely used in various fields, because they possess physical and chemical properties that qualify them to associate with many metal ions to form complexes that have proven useful in many practical applications in various fields [29]. In recent years, the work of researchers has focused on studying the effectiveness of this type of compound when used as catalysts in the fields of organic and biochemistry, and the effectiveness of the rules of XF may be attributed to the pair of non-egyptian hybrid electrons (SP²) For nitrogen corn azomethine group.

The rules of XV described many advantages, the most important of which is the relatively high stability and this stability is related to the raw materials necessary to prepare these rules (carbonyl and amine compounds) in terms of aromatic qualities. Compounds containing Arelian compensators have been characterized by high stability[30] relative to the XV rules derived from fibrous compounds, which are often solid and have relative thermal stability. While the rules for the recovery of derived from the compound of the Leviticus liquid qualities, the stability of the rules of the Schiff aromatic due to the resonance. The stability

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of Schiff's aromatic rules is due to the resonance status of the associated aggregates on both sides of the azomethine group. Heterocyclic compounds are also important because they are of great importance in multiple field. A Heterocyclic chemistry is one of the most complex branches of organic chemistry for the variety of its synthetic procedures and for its physiological and industrial significance [31] Heterocyclic compounds form the most extensive group of organic compounds and become more important Ever in full aspects of pure and applied chemistry. Heterocyclic compounds are known as cyclic compounds in which the ring contains one or

more atoms of a non-carbon element, the most commonly used being oxygen, nitrogen, and sulfur. The reason why oxygen, nitrogen and sulfur are more used in the formation of these compounds is that the angle of equivalence of these elements is very close to the angle of carbon equivalence, and then these atoms can replace the carbon atoms. The stability of these rings is good. Heterocyclic compounds may have three or more atoms, and heterocyclic atoms in the ring may be more than one atom. (32)

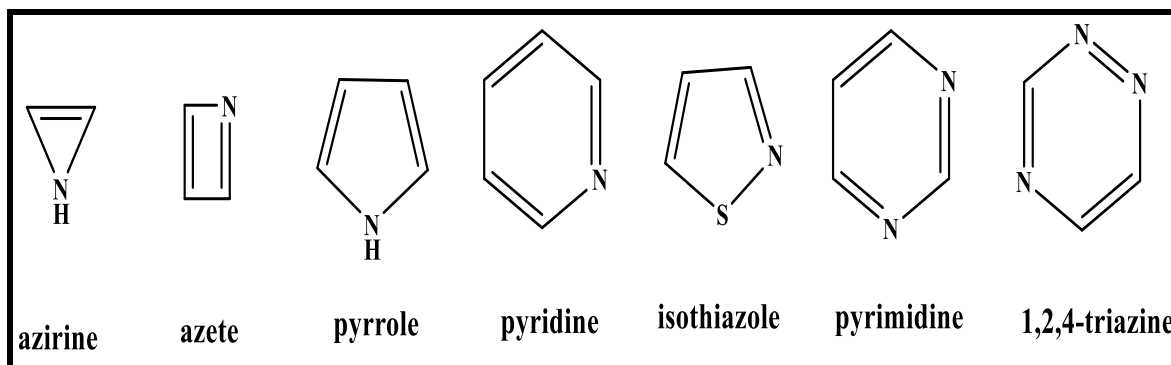


Figure 4: Heterocyclic compounds of different rings

Heterocyclic compounds are prepared by the preparation methods found in organic chemistry, as these compounds constitute the most important and largest varieties of organic compounds and include about 55% From compounds prepared in the field of organic chemistry [33] methods of preparation for these compounds have been developed using catalysts [34].

EXPERIMENTAL

Preparation of Azo Compounds (H1-H8) [35]

1. Dissolve (0.01 mole, 1.53g) of the 5-amino amylic acid compound in a mixture of (15% HCl) and cool the mixture to C°(0-5) .
2. Add the sodium nitrate solution (0.5 g sodium nitrate in 2.5ml distilled water) to the mix with stirring and note that the temperature does not rise from C°(0-5) after which leave the solution for (20-10) Minute to be diazonium salt
3. Dissolve (0.01 moles) of the compound containing carbonyl group (aldehyde or ketone) in 10% of sodium hydroxide)
4. Add the dezonium salt formed in step number (2) to step number (3) with continuous stirring after that leave the

solution for 30 minutes where the deposition then filters the mixture and wash the precipitate and re-crystallize. The reaction was followed up by thin-layer chromatography (TLC) using chloroform and ethanol in a ratio of (1:1) and table number (2-2) showing the prepared compounds and physical qualities of it.

DIAGNOSIS OF H1-H8 COMPOUNDS

Azo compounds (H1-H8) were prepared by converting 5-amino amylic acid into diazonium salt in the presence of sodium nitrite and hydrochloric acid and under the C° (5-0) temperature for nitroso (NO) Temperatures were controlled because the increase caused the disintegration of the diazonium salt and its decrease below the permissible limits will lead to the non-product and after we get the diazonium salt it was reactor with a number of aromatic-dyed compounds Or ketones to form the Azo compounds and the compounds of the blood and ketones such as:-
Vanillin, Salicylaldehyde, 4-Bromobenzaldehyde, 3-Nitrobenzaldehyde 4-Ethylbenzaldehyde, Acetophenone, 4-Aminoacetophenone, 4-Bromoaceto phenone as shown in the digram:

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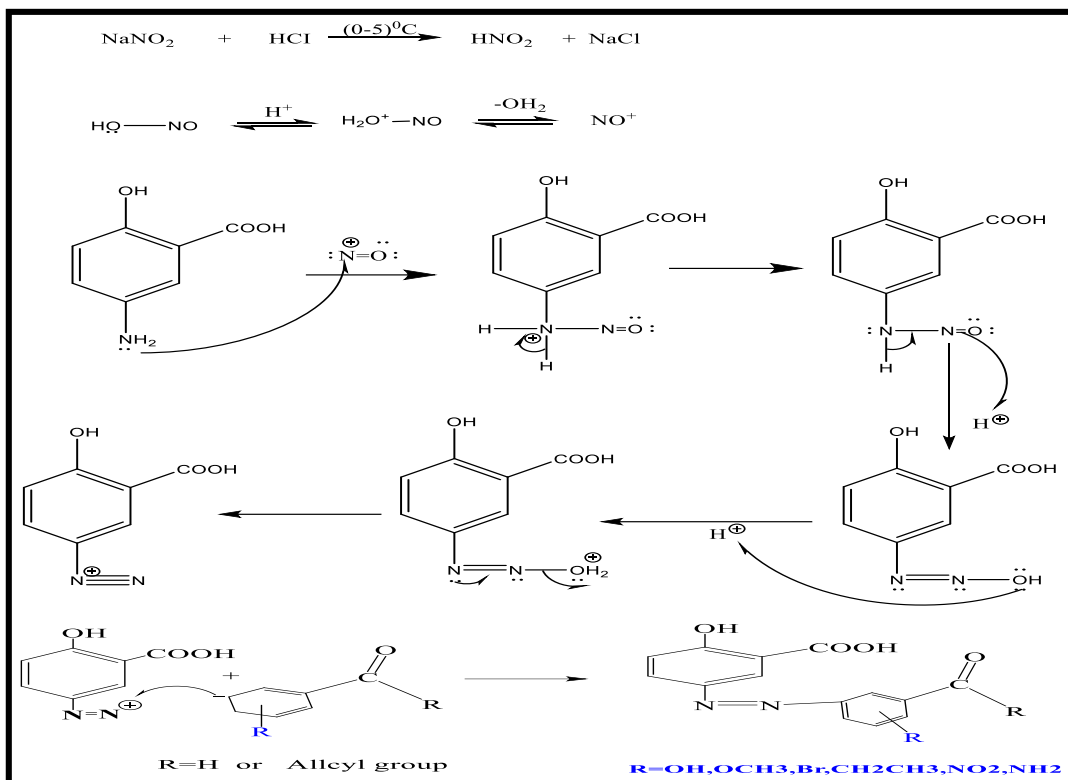


Figure 5: Mechanics of preparation of H1-H8 azo compounds

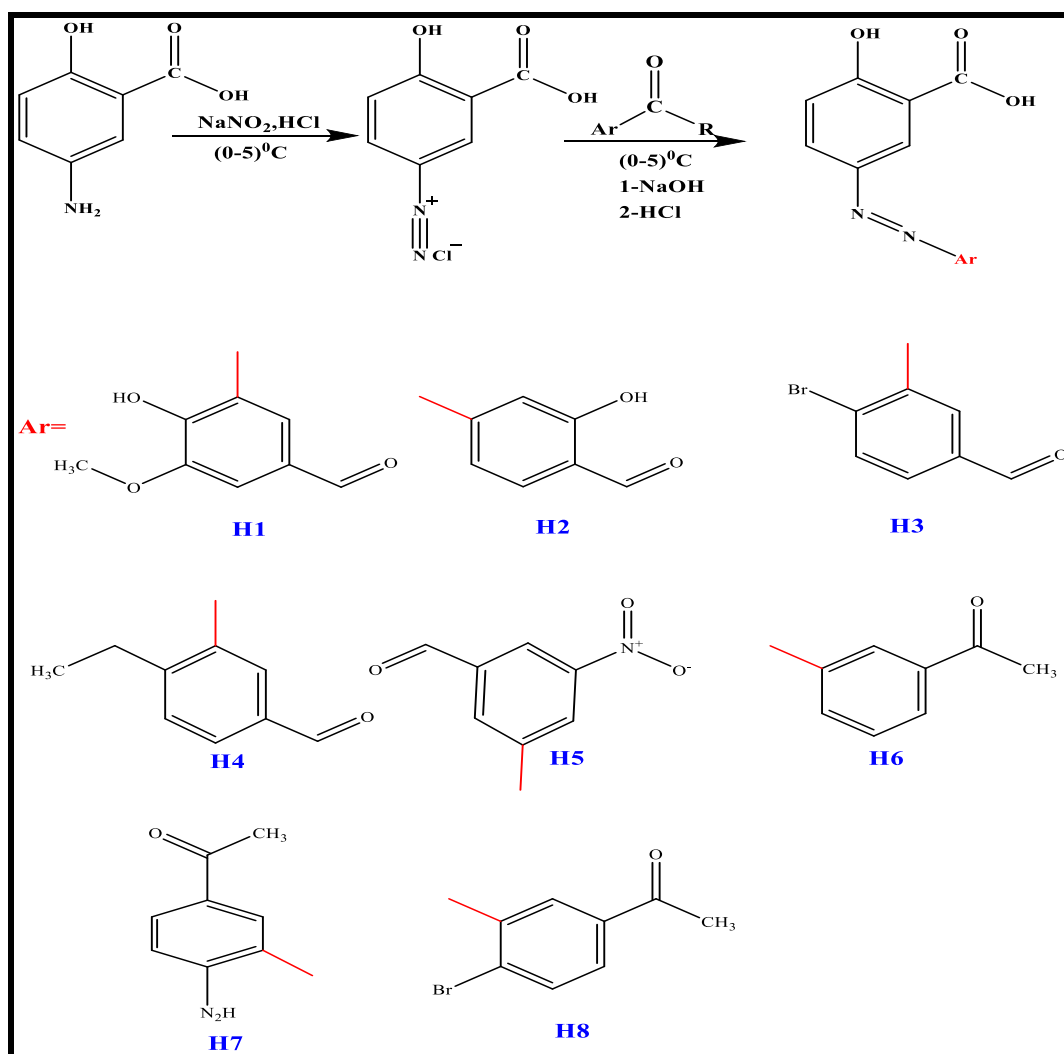
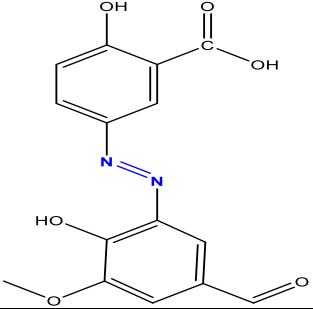
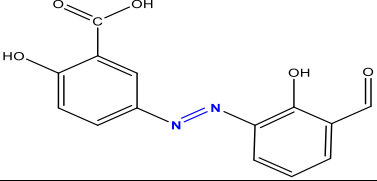


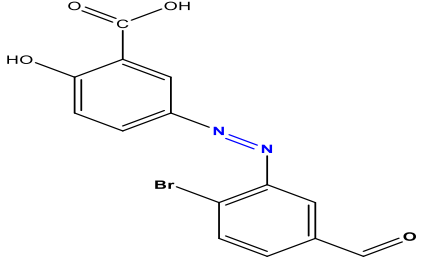
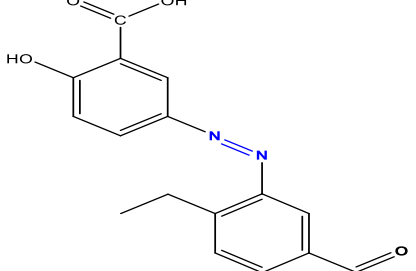
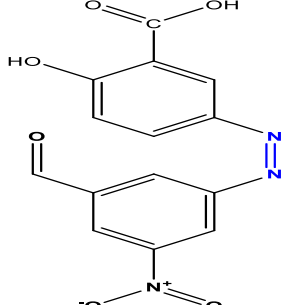
Figure 6: Prepare Azo H1-H8 compounds

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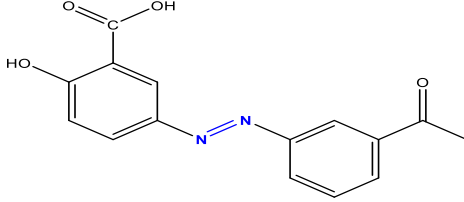
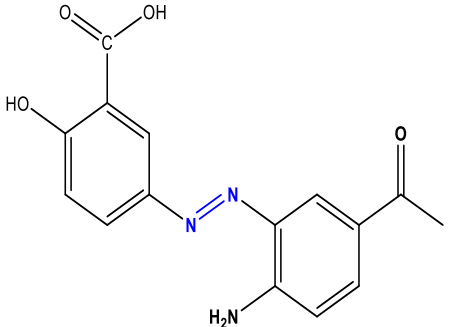
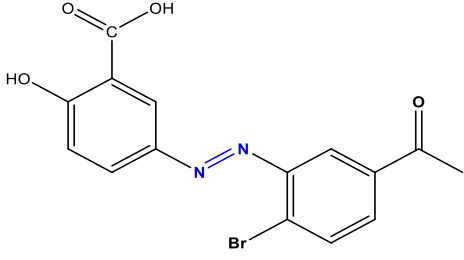
The potential reaction mechanism for the formation of Azo compounds containing an acetic (N=N) is in the figure 5.[36]

Table No1:- Physical Properties of Azo Compounds(H1-H8)

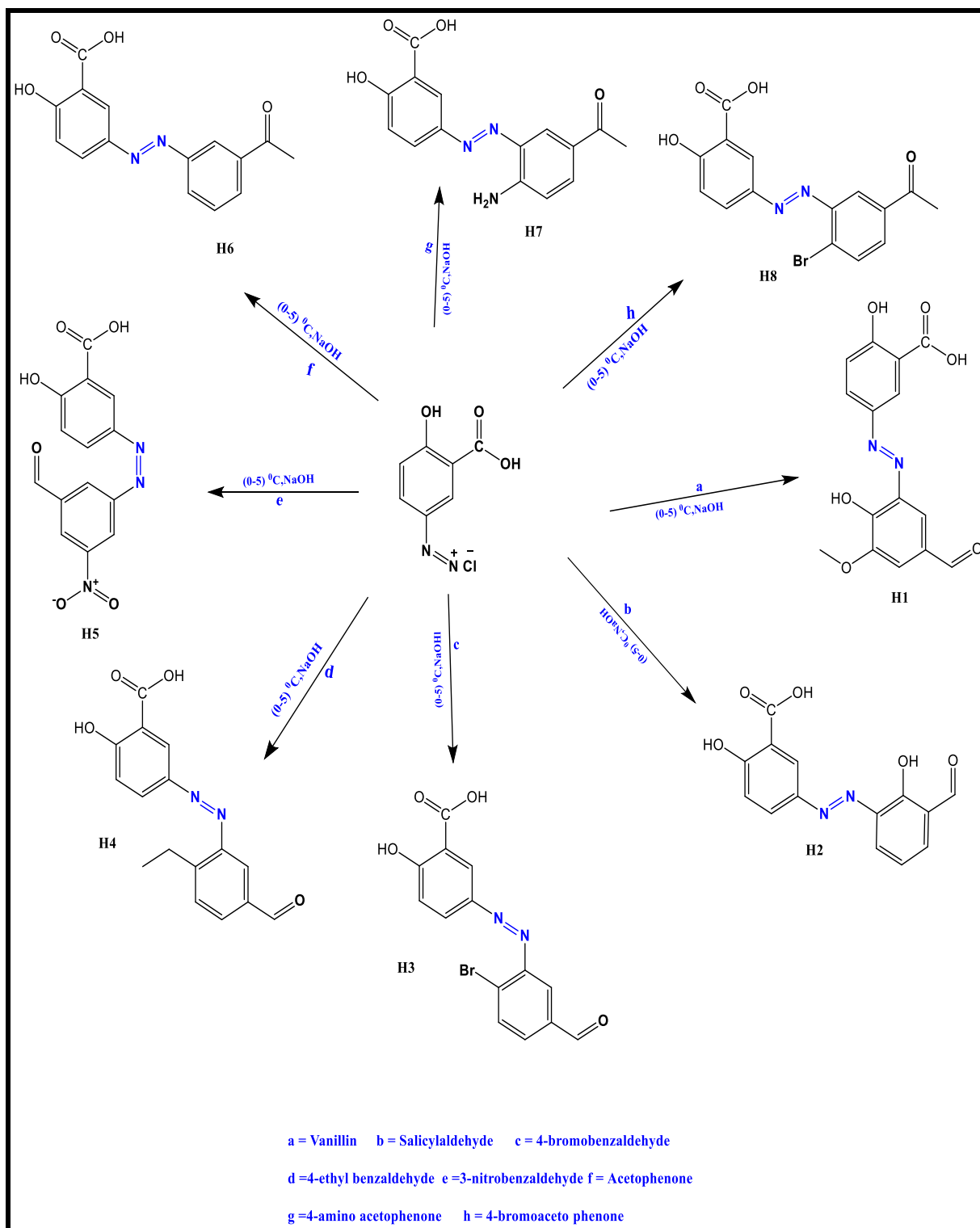
No. of compound	M.formula	Color	M.Wt	M.P	Yield %	Rf
H1		brown	316	125-127	74	.680
H2		Green-yellow	286	162-164	82	0.50

H3		brown	349	150-152	75	0.90
H4		Green-yellow	298	153-156	91	0.70
H5		brown	315	140-142	78	0.80

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H6		brown	284	165-167	81	0.40
H7		black	299	132-134	73	0.60
H8		brown	363	160-162	70	0.50

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Scheme1: preparation of azo compounds (H1-H8)

Antibacterial activity

Disk diffusion method was used to examine the antibacterial activity of heterocyclic compounds against Gram-positive bacteria (*Staphylococcus aureus*) and Gram-negative bacteria (*Escherichia coli*). Akkar dishes were sterilized with

autoclave at 121°C for 15 minutes., then the agar is poured onto the tablets and left to harden, then 4 holes (6 mm) are made. 0.1 mL of the prepared compounds solution (0.01 g in 2 ml of DMSO) is added in each hole, and these plates are incubated at (37 degrees Celsius) for (24 hours)[37]

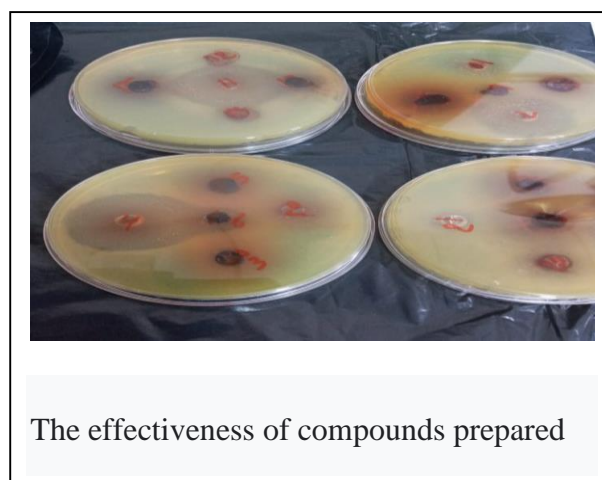
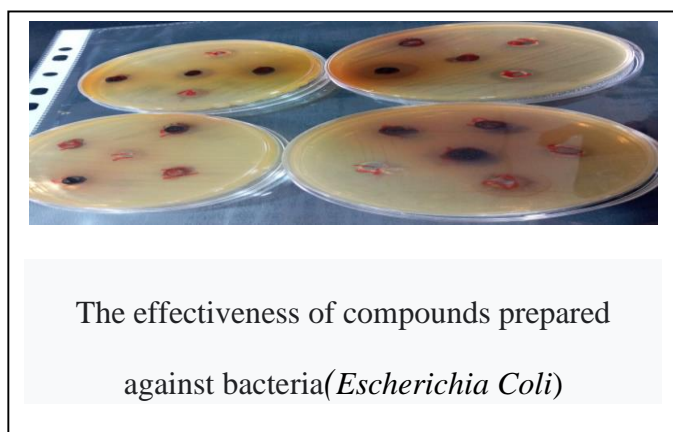
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Table No. 2: inhibition zone (mm) in the bacteria:-

Com.NO	Inhibition Zone (mm)	
	<i>E.Coli</i>	<i>Staph.</i>
H1	13	13
H2	13	16
H3	12	18
H4	16	18
H5	15	16
H6	12	17

Figure 7: The effectiveness of compounds prepared against bacteria

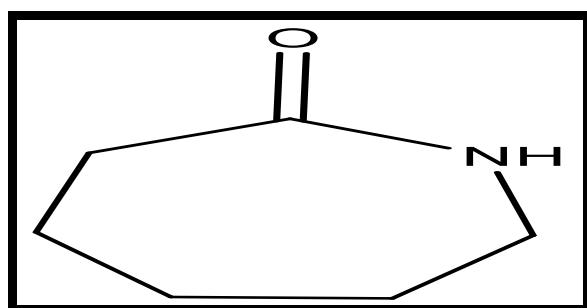
Importance of heterocyclic compounds:-



Heterocyclic compounds are of great importance, they are mainly involved in the formation of proteins, lipids, nucleic acids, chlorophylls, enzymes and vitamins, and more than 90 percent of drugs contain heterogeneous compounds. Which are considered biologically active as antifungal activity [38], anti-inflammatory [39], anti-bacterial [20], herbicide, anti-allergic, antispasmodic, and anti-cancer [21]

Heterocyclic compounds are used in the manufacture of certain drugs such as antibiotics (penicillin) and dyes such as

(xanthan dyes) and interfere with the synthesis of vitamins such as vitamin C (ascorbic acid) which contains the furan ring as well. In the manufacture of nylon used in the clothing industry such as (poly caprolactam) [30]. These compounds are the active group in many enzymes and co-enzymes [31,32] and most of these compounds extracted from animal and plant sources, these compounds can also be prepared in chemical laboratories [28].



caprolactam formula

RESULTS AND DISCUSSION

Azo compounds (H1-H8) were validated by conducting infrared spectrum analysis as well as the NMR spectrum and melting point measurement as well as the initial detection

conducted through TLC technology. Studying the FTIR spectrum of prepared compound (H1-H8), we found a new absorption peak at cm^{-1} 1483-1446 (returning to $\text{N}=\text{N}$) and a band appearing at cm^{-1} 1666 (1712-return to $\text{C}=\text{O}$) and the appearance of a band at cm^{-1} 3200-3568 going back to OH and the appearance of a band at cm^{-1} 1506-1591 (returning to $\text{C}=\text{C}$). Other evidence proving the correctness of the prepared compounds (H1-H8) is the complete disappearance of the two bands at cm^{-1} 3200-3500, which belong to the group of amines (NH_2).

The ^1H NMR spectrum of prepared compounds (H1-H8) measured using DMSO-d_6 as a solvent and the chemical displacement of the PHM unit was observed when multiple signals (multiplet) appeared at the site A ppm (6.23-9.8) due

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to aromatic ring protons and the appearance of a signal mono at ppm(12.04-12.2) returned to proton COOH group.

H1 was diagnosed using the infrared spectrum 'A new absorption package was observed at (cm-1 1464) belonging to the group (N=N) and the complete disappearance of the two packages belonging to the group of the Secretary(NH2) and this is evidence of the formation of Azo compounds that

To (C-H)Aromatic and the appearance of a bundle at (cm-11585) belongs to the group(C=C)as showninFigure(8).,:

contain On the Azo group in its composition after the disappearance of the amine group(NH2) as well as the appearance of a package at (cm-1 1666) belonging to the group (C=O) and the appearance of a wide package at (cm-1 3200-3650) return To the (OH) and the appearance of the pack when (cm-1 3124) returns

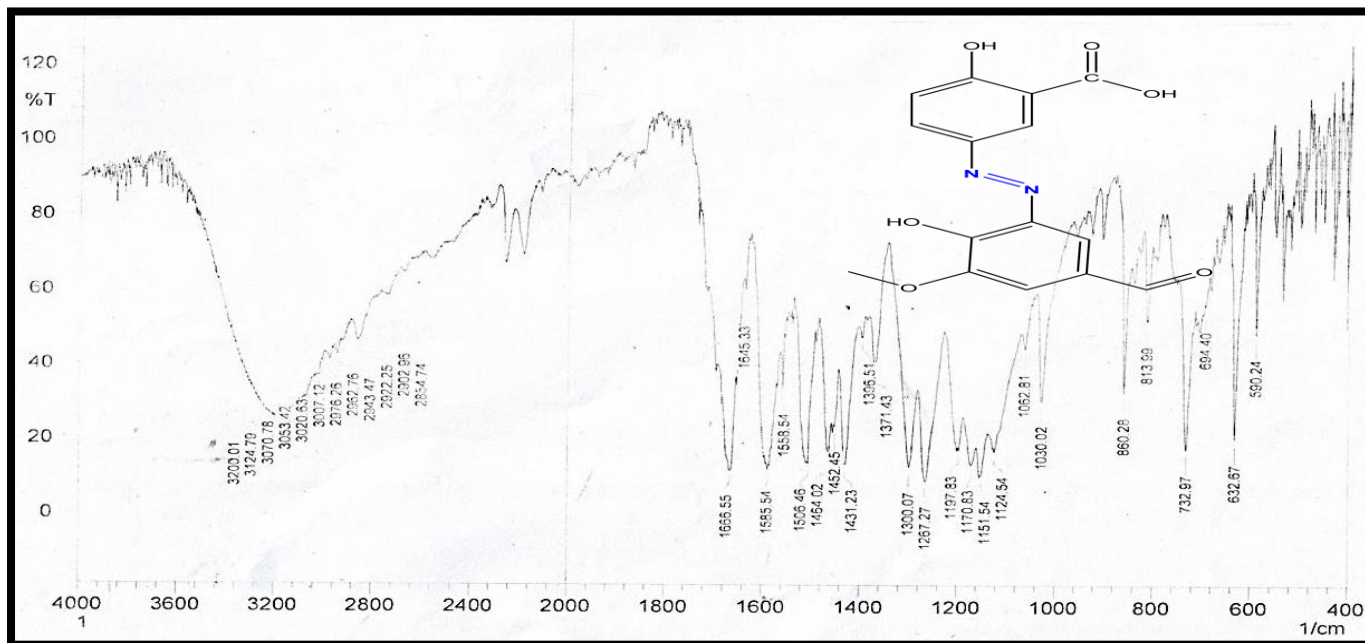


Figure 8:H1 for FTIR Spectrum

The FTIR spectrum of the H2 compound as in Figure(9) We note the emergence of a new absorption pack at cm-1 1477 going back to (N=N) and the appearance of the pack at cm-1 1708 going back to (C=O) and the emergence of Package at -2500 cm-13543 goes back to (OH) and the appearance of a

pack at cm-1 3107 goes back to (C-H) Aromatic and the appearance of a pack at cm-11545 goes back to (C=C).

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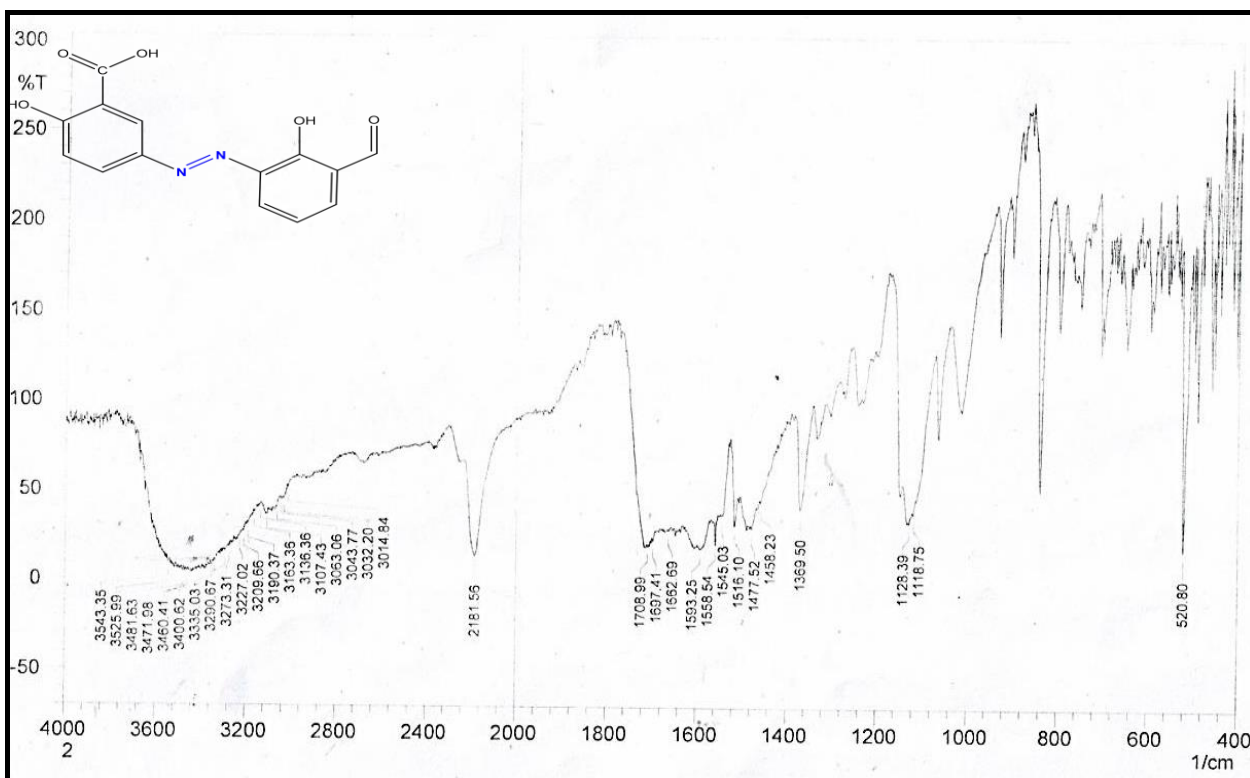


Figure 9:H2 for FTIR Spectrum

The FTIR spectrum of the H3 compound as in Figure(10) We note the emergence of a new absorption pack at (cm-1 1446) going back to (N=N) and the appearance of a pack at (cm-1 1708) going back to (C=O) The appearance of a bundle at(-2500cm-13468) dates back to (OH) and the appearance of a bundle at (cm-1 3084) going back to (C-H)Aromatic and the appearance of a bundle at (cm-11585) going back to (C=C) and the appearance of Pack at (cm-1650) goes back to (C-Br).

The ¹HNMR spectrum of the H3 compound as in Figure (11) was observed as a single signal at (9.2pm) returning to the Aldehyde group proton and the appearance of a single signal at (pm12.04) returning to the COH group proton and as such appearing multiple signals At ppm(7.02-9.8) you return to the protons of the aromatic ring.

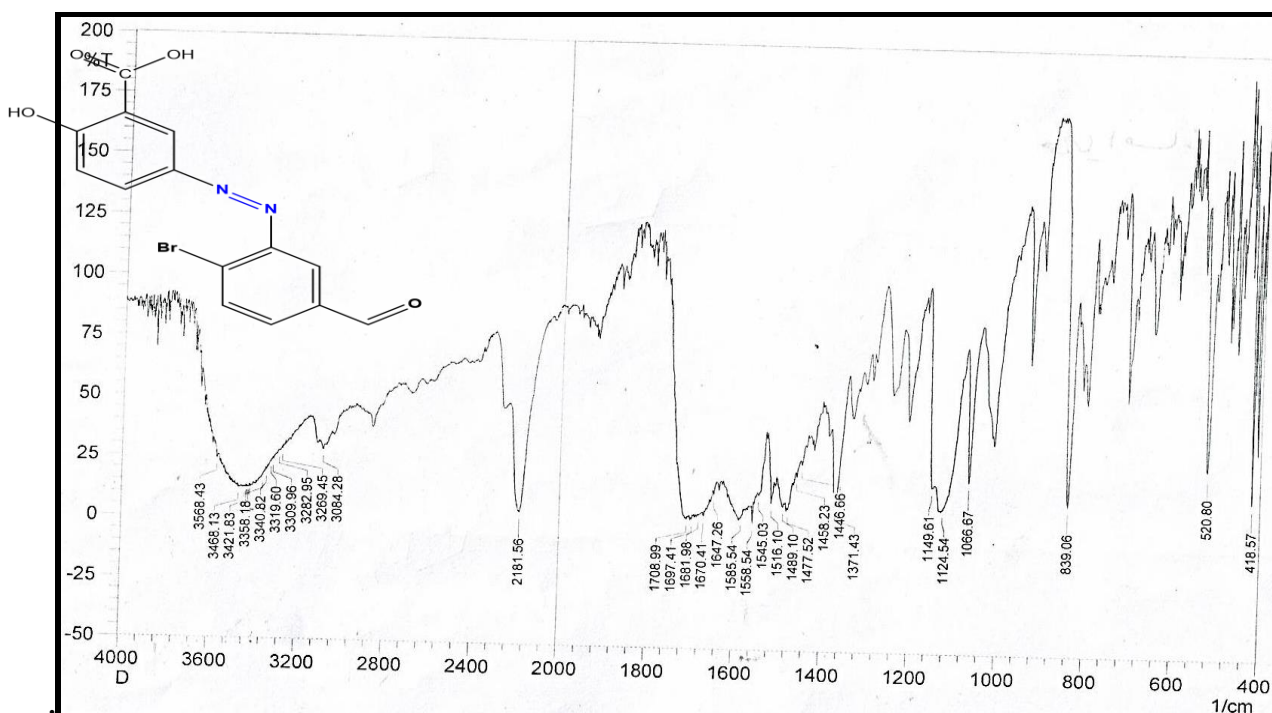


Figure 10:H3 for FTIR Spectrum

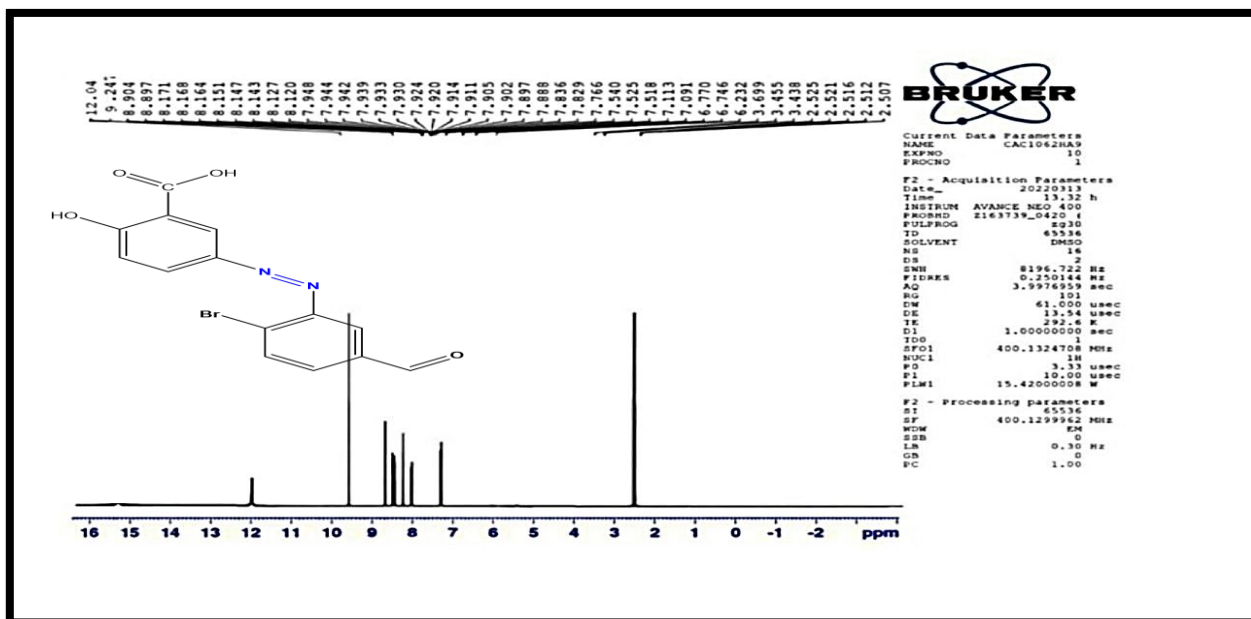


Figure 11:H3 Compound 1HNMR Spectrum

The FTIR spectrum of the H4 compound as in Figure (12) We note the emergence of a new absorption pack at cm^{-1} 1477 going back to (N=N) and the emergence of a pack at cm^{-1} 1708 going back to (C=O) and the emergence of Package at - The 1HNMR spectrum of the H4 compound as in Figure(13) was observed as having a single signal at (pm 12.04) going back to the COOH group proton and a triple signal appearing at (1.18pm) and another quad signal at (2.52pm) returning to

2500 cm^{-1} -13527 Dating to (OH) The appearance of the package at cm^{-1} 3072 is due to (C-H) Aromatic and the appearance of the package at cm^{-1} 1545 is due to (C=C).

the totals of the instance and the appearance of a single signal at (9.8 ppm) belong to the proton group Aldehyde and also the appearance of multiple signals at the ppm(7.09-9.8) belong to the protons of the aromatic ring

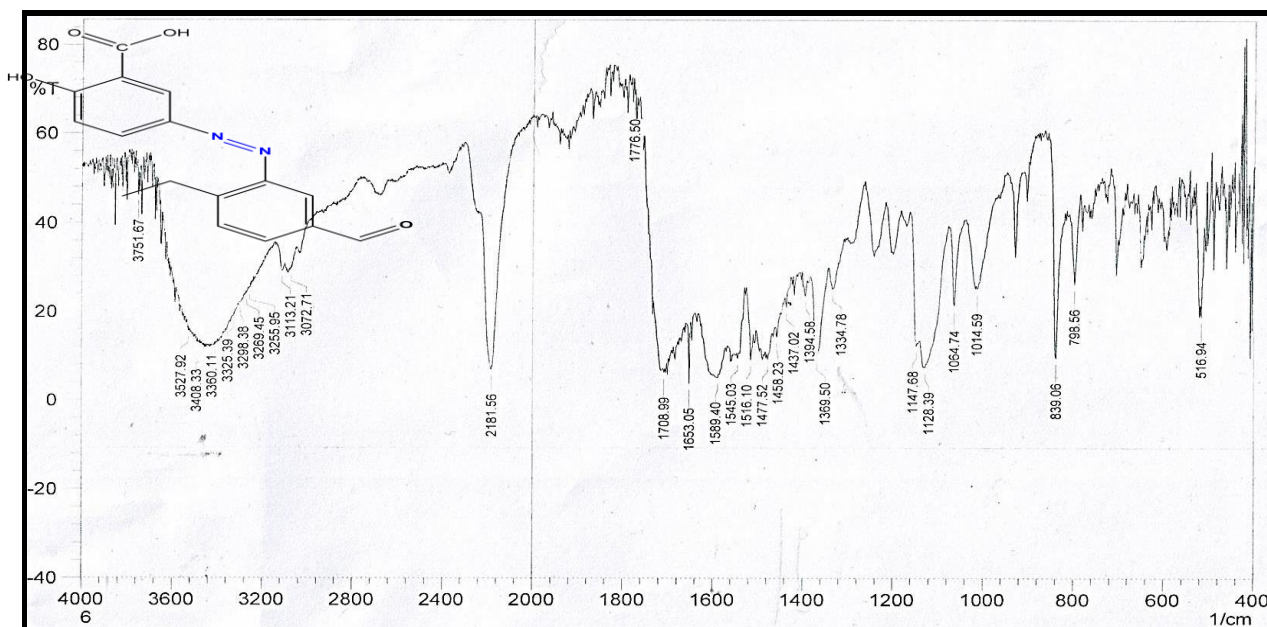


Figure 12:H4 for FTIR Spectrum

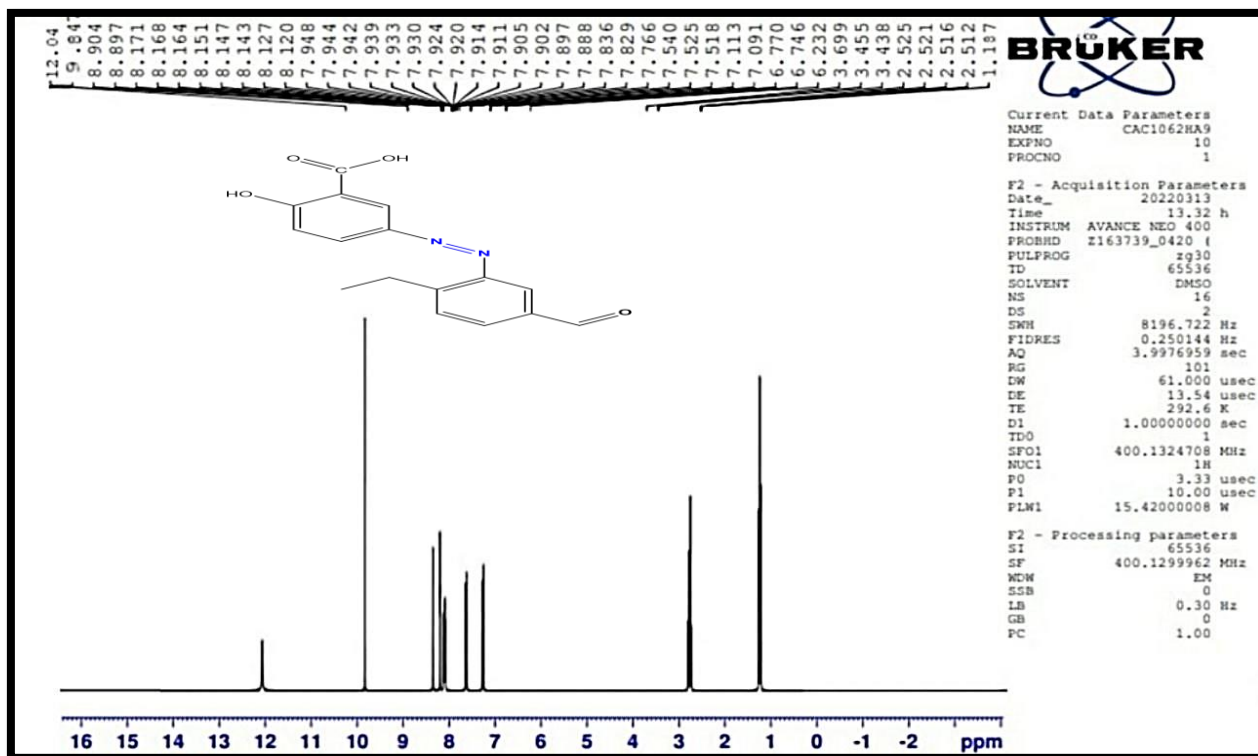


Figure 13: H4 Compound 1HNMR Spectrum

The FTIR spectrum of the H5 compound as in figure(14) We note the emergence of a new absorption pack at cm-1 1483 going back to (N=N) and the appearance of the pack at cm-1 1707 going back to (C=O) and the emergence of Package at -2500cm-13452 dates back to (OH) and the appearance of a package at cm-1 3086 goes back to (C-H)Aromatic and the

appearance of a package at cm-11591 goes back to (C=C).Spectrum 1HNMR of the compound (H5) as in figure(15) Single-signal is observed at 1PM)12.04(due to Proton COH group and single-signal appearance at BM) (9.4 due to Proton Aldehyde group and such emergence Multiple signals at ppm(6.23 -9.8) are due to aromatic ring protons

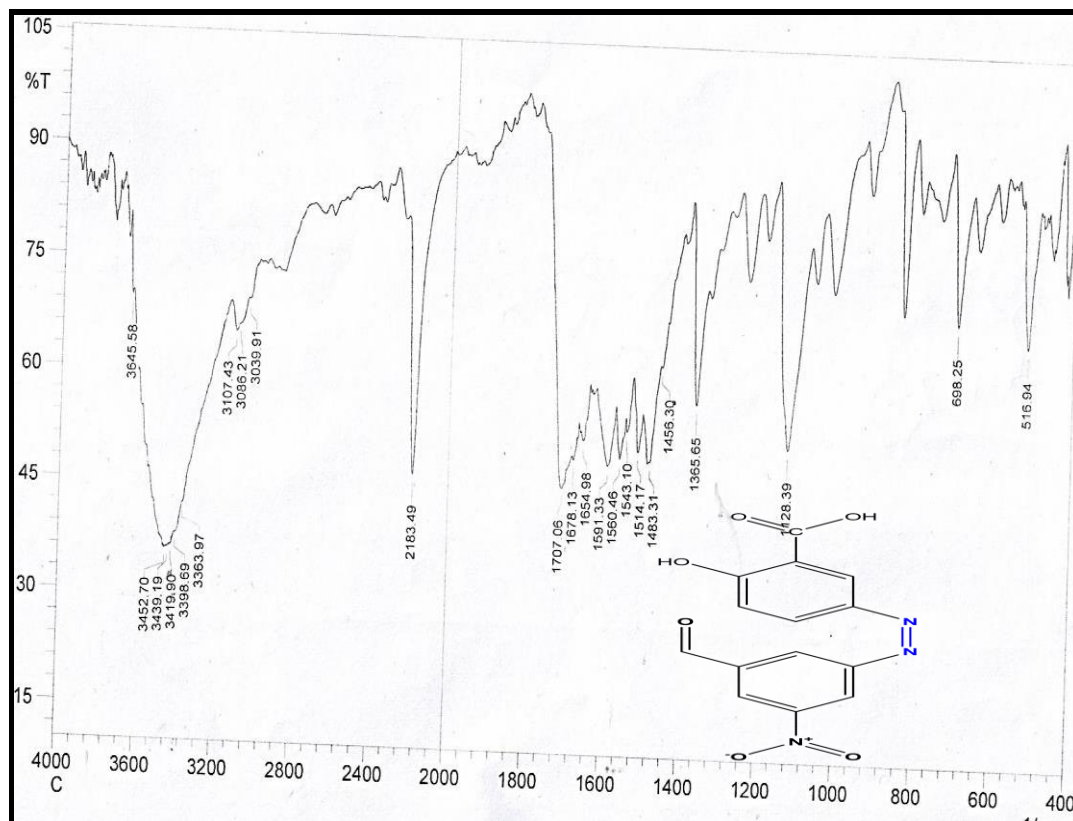


Figure 14: H5 for FTIR Spectrum

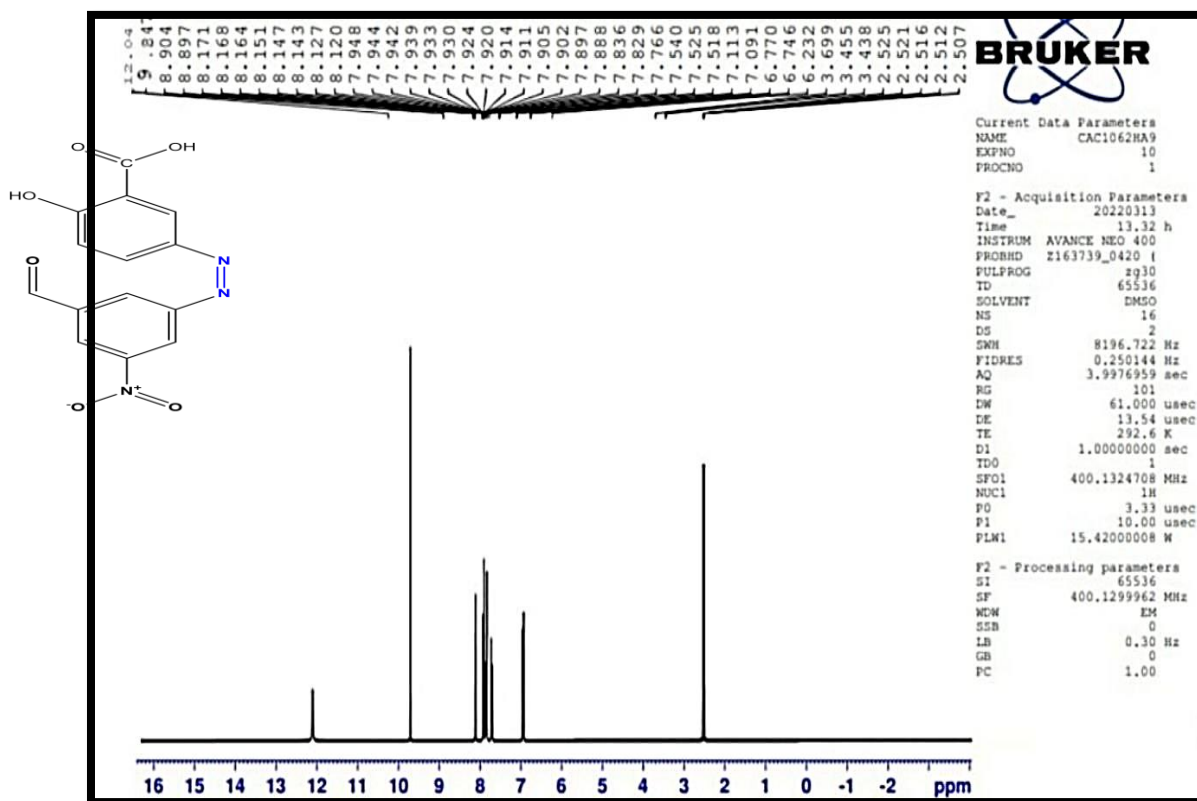


Figure 15: H5 For Compound 1HNMR Spectrum

The FTIR spectrum of the H6 compound as in Figure(16) We note the emergence of a new absorption pack at cm^{-1} 1477 going back to (N=N) and the appearance of the pack at cm^{-1} 1712 going back to (C=O) and the emergence of Package at cm^{-1} 2500-3454 goes back to (OH) and the appearance of a package at cm^{-1} 11589 goes back to (C=C).

Spectrum 1HNMR of the compound (H6) as in figure(17) Single-signal is observed at 4PM)12.2 (due to Proton COH group and signal appears at PM)2.5 (DMSO-d6 returns, as do multiple signals appear at ppm(7.09-9.8) Returns to the protons of the aromatic ring .

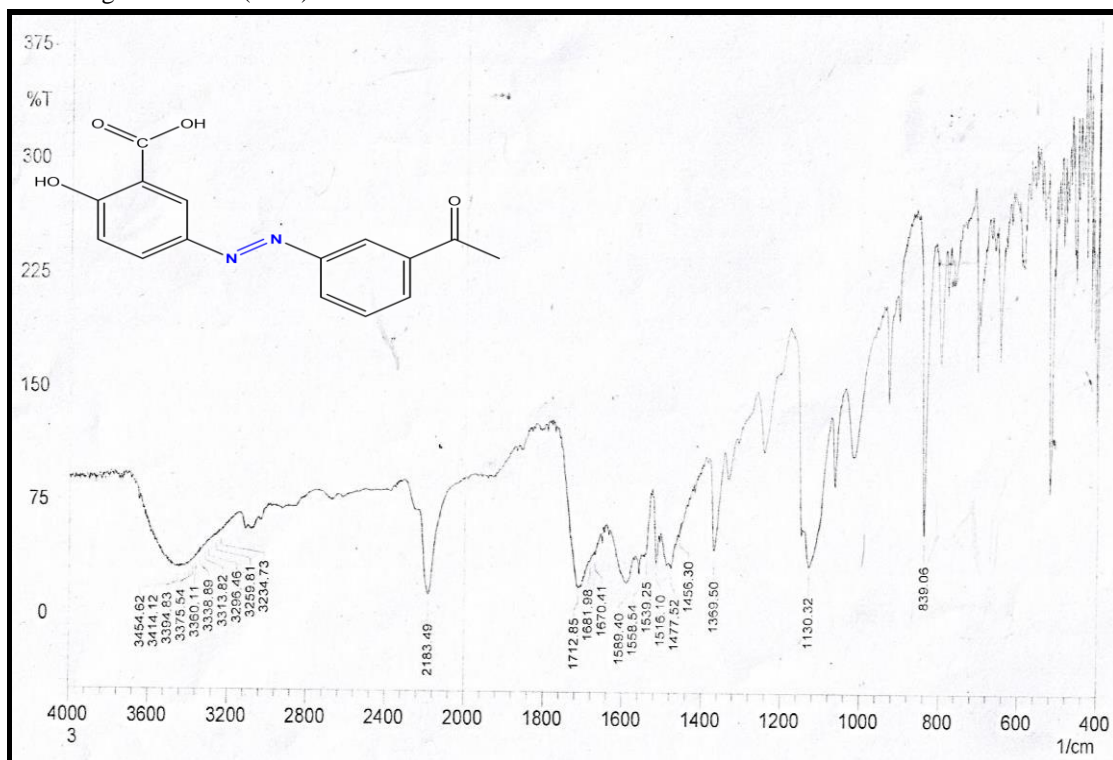


Figure 16: H6 for FTIR Spectrum

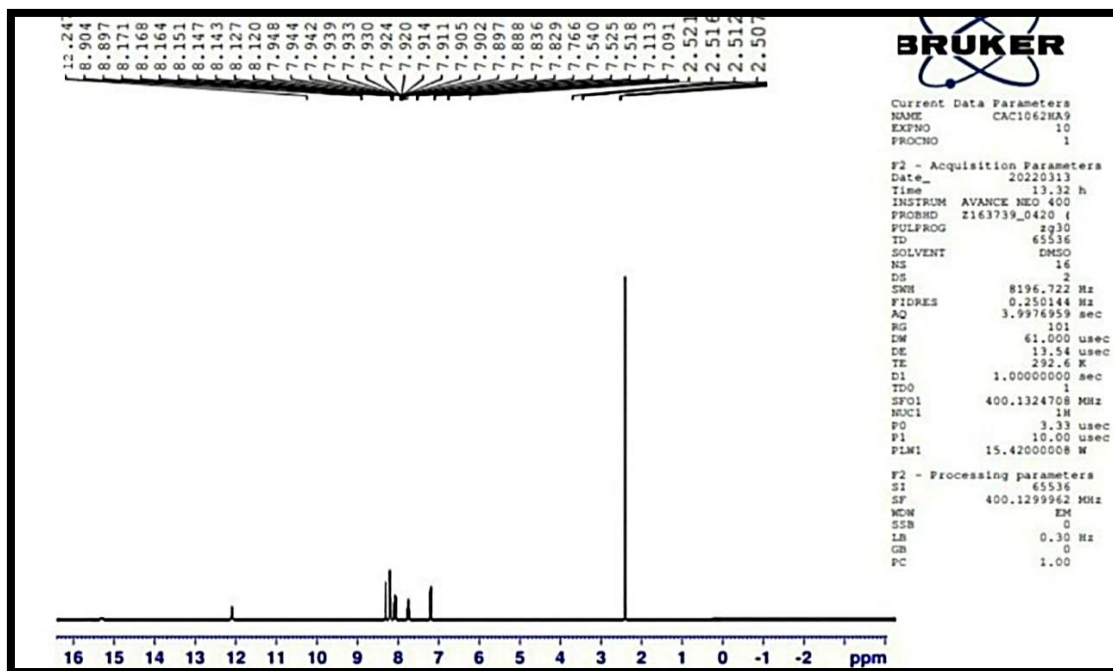


Figure 17: H6 For Compound1HNMR Spectrum

The FTIR spectrum of the H7 compound as in figure(18) We note the emergence of a new absorption pack at cm-1 1514 going back to (N=N) and the appearance of the pack at cm-1 1678 going back to (C=O) and the emergence of Pack when(cm-13441,3335) returns to (NH₂) and the appearance of the pack at -2500cm-13225 goes back to (OH) and the appearance of the pack at cm-1 3063 goes back to (C-H)aromatics and the appearance of the pack at cm-1 2895 It protons.

dates back to (C-H)Fatty and the emergence of a pack at cm-11560 going back to (C=C).

Spectrum 1HNMR of the compound (H7) as in Figure(19) One signal is observed at 12.04(returns to Proton COOH Group) and a binary signal appears at (5.12ppm) return to NH₂ Group as well as multiple signals at the ppm (7.09 - 9.8) Back to aromatic ring

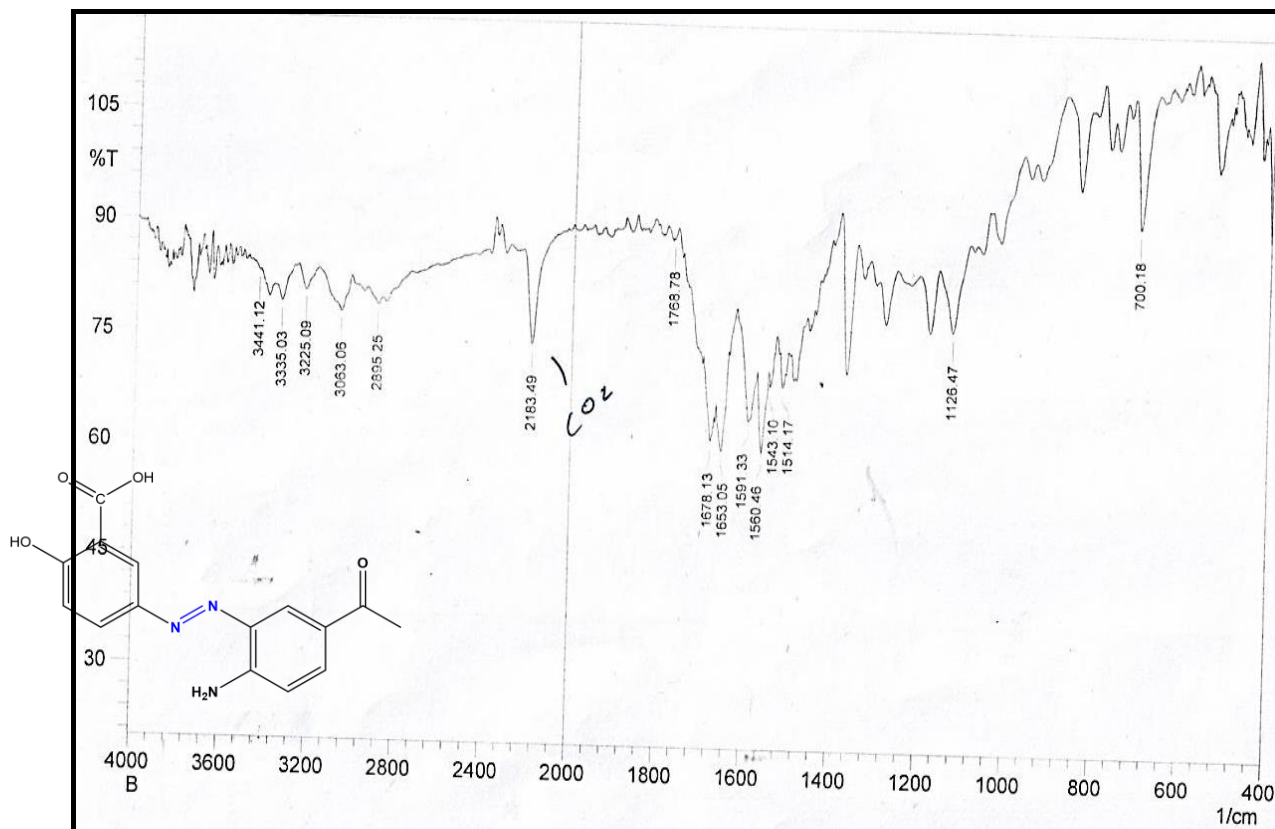


Figure 18: H7 for FTIR

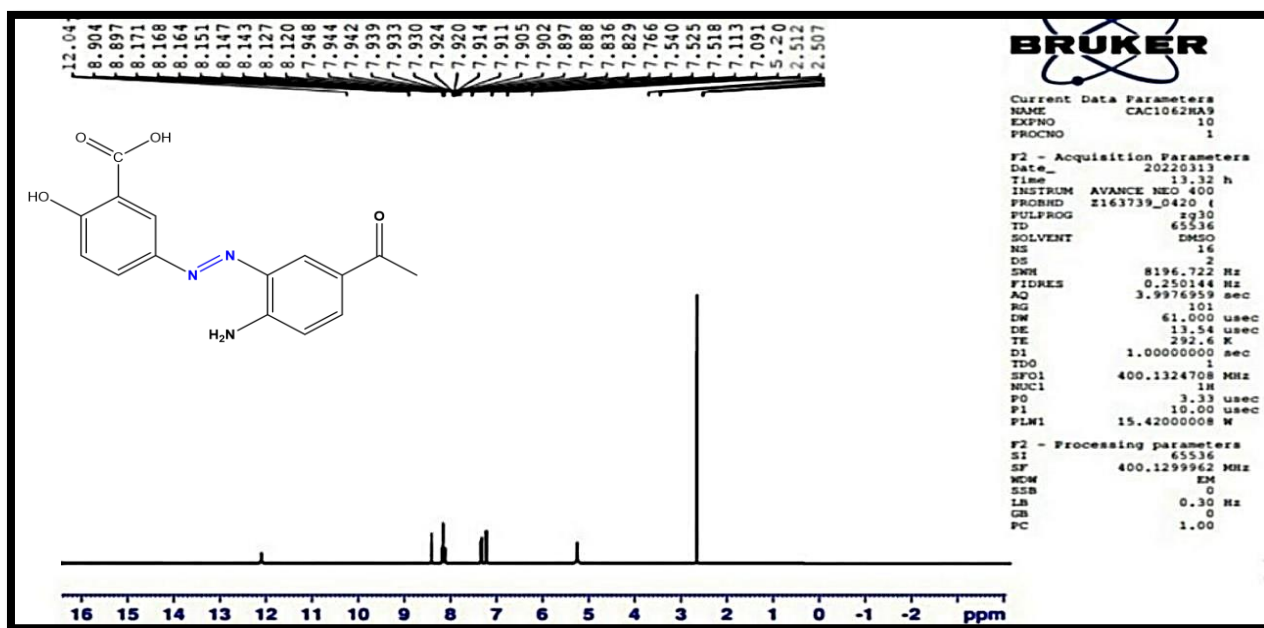


Figure 19: H7 compound FTIR spectrum

The FTIR spectrum of the H8 compound as in figure(20) We note the emergence of a new absorption pack at cm-1 1477 going back to (N=N) and the appearance of the pack at cm-1

1712 going back to (C=O) and the emergence of Package at -2500cm-1 3200Due to (OH).The appearance of the package at cm-11585Due to (C=C).

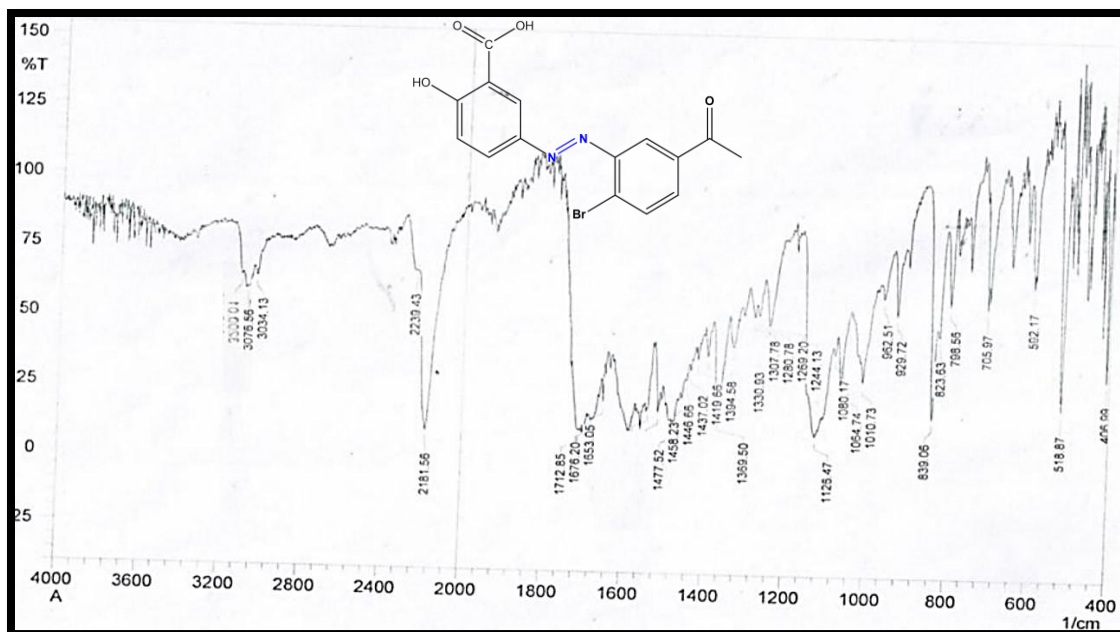


Figure 20: H8 for FTIR

CONCLUSIONS:

In this study, new derivatives of 5-amino-amselic acid carrying different heterocyclic compounds were prepared and diagnosed, with some applications being studied, and the conclusions can be summarized as follows:

- The possibility of preparing the Azo compounds by converting 5-amino amylic acid into dizonium salt in the presence of sodium nitrite and hydrochloric acid and under the temperature of C⁰ (5-0) to obtain nitroso (NO) and after we get on the dezonium salt it was reactor with

a number of aromatic, aldehyde or ketone compounds to form azo compounds.

- The possibility of preparing compound of Schiff bases as well as Azo compounds and heterocyclic compounds of 5-amino amelic acid pure and high output ratio.
- Schiff base compound as well as Azo and heterocyclic compounds prepared in the research are stable because of their non-disintegration or discoloration due to ambient weather conditions and their high melting point

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