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Synthesis of some new oxazepine compounds derived from cyanoethyl acetate and study their inhibitory activity against some pathogenic bacterial species

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***Abstract*---**Involves synthesis of Schiff bases (hydrazone) by the reaction of acid hydrazide(D_1) (which is prepared from the reaction of cyanoethyl acetate with hydrazine hydrate at (85%) with aromatic aldehydes to obtain compounds (D_{2-6}) and by the reaction of compounds (D_{2-6}) with phthalic anhydride Compounds (D_{7-11}) are obtained, the biological activity of the prepared compounds is studied through two types of Gram-positive and Gram-negative bacteria Staph. aureus, Escherichia Coli and Compare it with one type of antibiotic (Gentamycin). The validity of the structures is confirmed by spectroscopic methods (FTIR, ^{1}H-NMR, ^{13}C-NMR, MASS). The disk diffusion assay technique was used to investigate the antibacterial activity in vitro against two types of bacteria: gram-positive and gram harmful bacteria. The minimum inhibitory concentration [MIC] was measured using standard medications. The findings revealed that azetidine derivatives outperformed drugs in inhibiting the growth of both gram-positive and gram-negative bacteria.

***Keywords*---**Schiff bases (hydrazone), Oxazepine, Antibacterial activity.

1. Introduction

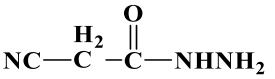
Schiff's bases are organic compounds that have in their composition azomethine group (CH=N) [1]. Schiff's bases are among the compounds that have been widely studied because of their chemical efficacy, physical properties and applications in many fields, including in drug chemistry and pharmaceutical industries, due to their biological activity to contain these compounds on Azomethine group, for this reason it is used as an antiviral for many diseases, such as antibacterial [2], antifungal [3], and that it is effective against viruses (Antiviral Activity) [4], and some Schiff's anti-malarial bases were used. malaria) [5], others have high activity against cancer cells [6], there are many Schiff bases have biological antimicrobial activity [7], there are many Schiff bases have anti-inflammatory and analgesic activity [8]. A heterocyclic unsaturated containing five carbon atoms, and there are three isomers of Oxazepine compounds. Oxazepine compounds and derivatives are of wide interest due to their wide spectrum of biological and pharmacological activities. Most of the oxazepine derivatives are used in wide medical fields such as antibacterials, anti-hypnotic muscle relaxants, anti-inflammatory and anti-epileptics [9], and antifungals [10], and there are some oxazepine derivatives that act as biological antigens for patients with leukemia and breast cancer [11], and some oxazepine derivatives have shown high efficacy against bacteria such as Staphylococcus aureus, Escherichia Coli**.**

2. Experimental

Melting points were obtained using an uncorrected Electro thermal Apparatus. FTIR model 84005 Shimadzu Japan. KBr disk infrared spectrophotometer Ultra shield 300 Mhz was used to record ^{1}H-NMR, ^{13}C-NMR, and MASS spectra. al-albaet university Jordan, Bruker 2003. If you're looking for a unique.

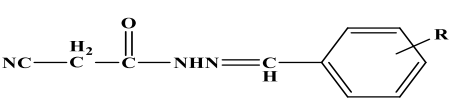
2.1. Synthesis of Cyanoacetohydrazide(D_1) [12,13]

In 20ml of Ethanol Absolute, a combination of (0.05 mol, 2.5 ml) cyanoethyl acetate, and (2.5 ml) Hydrazine hydrate (80%) was reflexed for (3) hours, then concentrated and chilled. The product was filtered and recrystallized by ethanol to produce brown with a M.P. of 87 percent (108-110 M).



2.2. Synthesis of hydrazone derivatives ($D_{2\text{-}6}$) [14,15]

To a solution of (0.02 mol) substituted benzaldehyde in (20ml) of absolute ethanol and then add drops of glacial acetic acid to the mixture, and add (0.02 mol) to it acid hydrazide dissolved in (25ml) absolute ethanol, then refluxed the mixture for 4 hours and cool the solution, and then filter and wash with distilled water and recrystallized the product with absolute ethanol the physical properties of the synthesized compound are given in Table(1).



2.3. Preparation of derivatives of 1,3-oxazepine 4,7-dione (D_{7-11}) [16,17]

The solvent was evaporated by distillation after a combination of (0.01mol) from Shiff base(D2-6) in (20ml) of dry benzene with (0.01mol) of phthalic anhydride was reflexed for (8) hours. The debris was mixed up with the impact ice. Filtration was used to separate the product, then re-crystallized to remove the Dioxins. Table (1) shows the physical characteristics of produced substances.

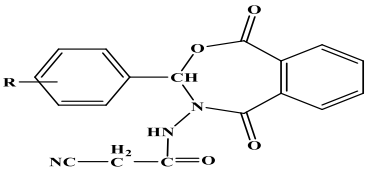


Table 1: Some physical properties of derivatives compounds (D_{2-11})

Comp. No.	R	Molecular Formula	M.Wt g/mol	Color	M.P. (^{0}C)	Yield (%)
D_2	2,3-Cl	$C_{10}H_7N_3OCl_2$	256.09	Yellow	140-142	83
D_3	2,4-Cl	$C_{10}H_7N_3OCl_2$	256.09	Golden yellow	147-149	85
D_4	4-Cl	$C_{10}H_8N_3OCl$	221.64	Yellow	138-140	79
D_5	4-OCH_3	$C_{11}H_{11}N_3O_2$	217.23	Orang	150-152	82
D_6	4-NO_2	$C_{10}H_8N_4O_3$	232.20	Brown	146-148	67
D_7	2,3-Cl	$C_{18}H_{11}N_3O_4Cl_2$	04.20	Light yellow	177-179	66
D_8	2,4-Cl	$C_{18}H_{11}N_3O_4Cl_2$	404.20	Yellow	189-191	63
D_9	4-Cl	$C_{18}H_{12}N_3O_4Cl$	369.76	Orang	167-169	68
D_{10}	4-OCH_3	$C_{19}H_{15}N_3O_5$	365.35	Light orang	165-167	58
D_{11}	4-NO_2	$C_{18}H_{12}N_4O_6$	380.32	Brown	175-177	53

3. Results and Discussion

The synthesis of Cyanoacetohydrazide from reaction (cyanoethyl acetate with hydrazine hydrate at 85) the FTIR spectra of compound D_1 showed two bands were observed at (3348, 3192) cm^{-1} belonging to (NH_2), and the appearance of absorption bands at (3136) cm^{-1} due to the stretching of (NH) bond. Also, two absorption bands appeared at (2980 and 2862) cm^{-1} due to the stretching of (CH_2) bond, in addition to the appearance of an absorption band at (2260) cm^{-1} due to the stretching of (CN) bond, and an absorption band appeared at (1703) cm^{-1} is due to the stretching of (CO) bond, and an absorption band appeared at (1255) cm^{-1} due to the stretching of (C-N) bond, and an absorption band appeared at (1105)cm^{-1} that was due to the stretching of (N-N) bond, as shown in figure (1), as these packages were close to what is found in the literature [18, 19].

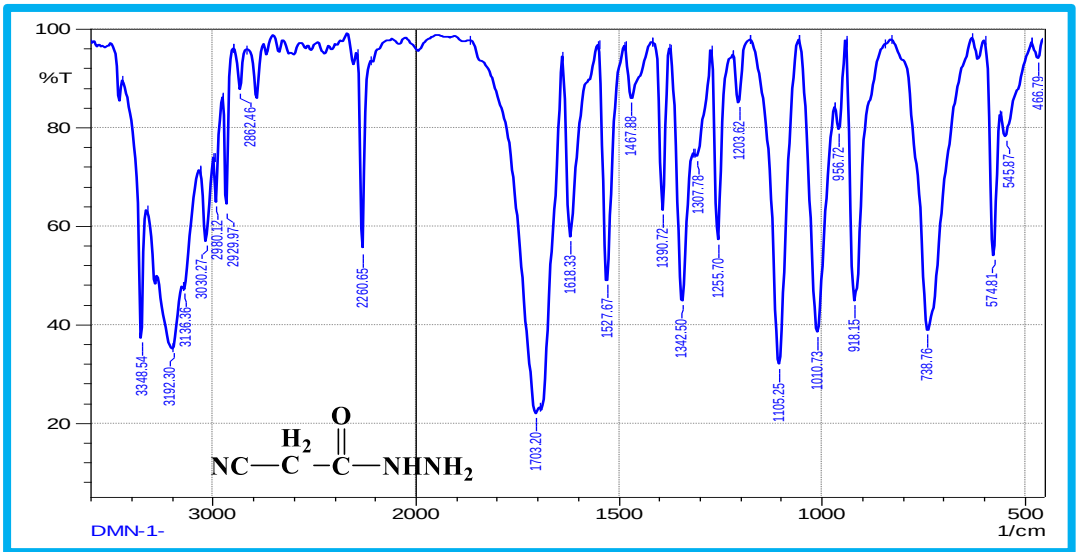


Figure 1: FTIR spectrum of D1

The nuclear magnetic resonance spectrum of the proton of the compound [D1] using a solvent (DMSO-d^6), shwed that a triple signal appeared at the position (5.75, 5.76, 5.77) ppm attributed to the proton of the (NH) and the appearance of a binary signal at the site (4.40, 4.41) ppm refers to the protonation of the amine group (NH_2), as well as the appearance of a signal at the site (4.28) ppm attributed to water protons, and the appearance of a single signal at the position (3.43) ppm attributed to the protonation of the (CH_2) group, and the appearance of a signal At position (2.39, 2.40, 2.41) ppm attributed to the protonation of (DMSO-d^6) [20, 21], as in figure (2).

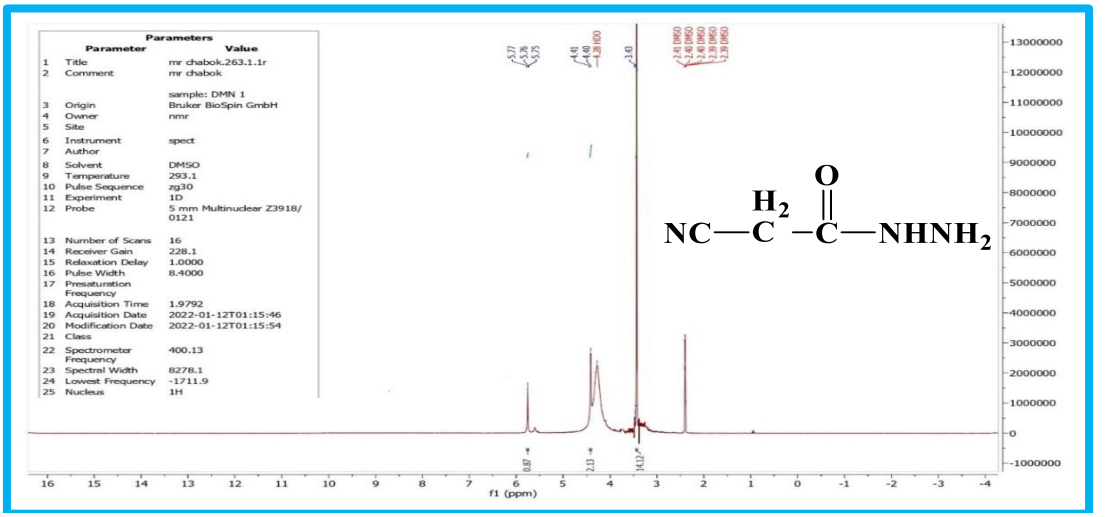


Figure 2: ^{1}H-NMR spectrum of D1

The nuclear magnetic resonance spectrum of carbon for compound [D1] using a solvent (DMSO-d^6), shwed that a signal appeared at the position (24.10) ppm

attributed to the carbon (CH_2) group, and a signal appeared at the position (116.50) ppm that was related to carbon. (CN) group, as well as the appearance of a signal at the site (162.34) ppm attributed to the carbon group (CO), and the appearance of signals at the site (39.08-40.34) ppm attributed to the solvent carbonate (DMSO-d^6) [22, 23], and as in figure (3).

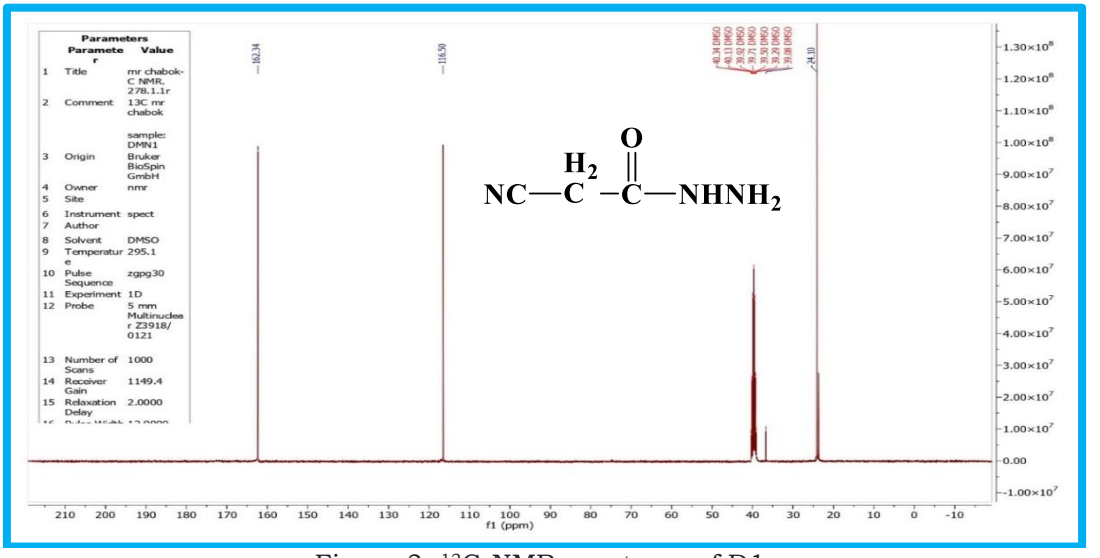
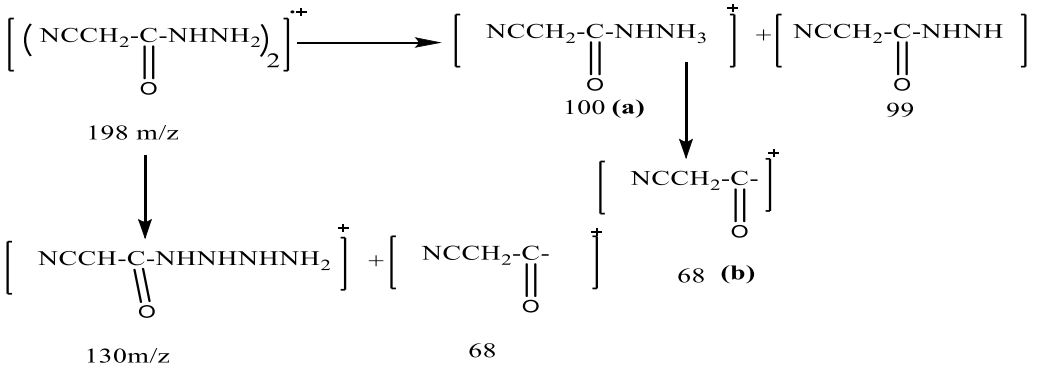


Figure 3: ^{13}C-NMR spectrum of D1

The mass spectrum of compound (D1) in (Figure 4) showed a peak at 198.8 observations that belong to the dimer form of the hydrazide molecule of molecular weight (99). The spectrum also showed a base peak at 100 observations [$NCCH_2$-C=O-$NHNH_3$]$^+$ and a peak appeared at 68 observations of the carbonyl group [$NCCH_2$-C=O]$^+$ [24, 25].



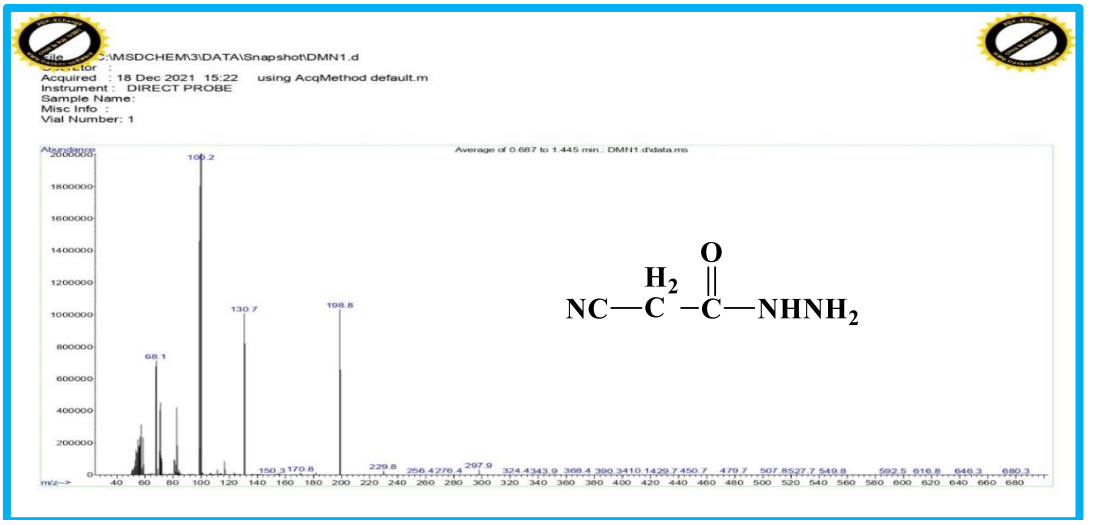


Figure 4: Mass spectrum of D1

The synthesis of Schiff bases from reaction (substituted benzaldehyde with acid hydrazide), the FTIR spectra of compound D_5 showed, it was noticed that the two bands of the amine group (NH_2) disappeared, and absorption bands appeared at frequency (3217) cm^{-1} due to the stretching of the (NH) bond, and the absorption band appeared at frequency (3090) cm^{-1} This is due to the stretching of the aromatic (CH) bond, as well as the appearance of two absorption bands at frequency (2918, 2835) cm^{-1} due to the stretching of the (CH_3) bond, in addition to the appearance of an absorption band at frequency (2258)cm^{-1} due to the stretching of the (CN) bond. An absorption band appeared at the frequency (1689) cm^{-1} due to the stretching of the azomethine bond (C=N), Also, an absorption band appeared at the frequency (1672) cm^{-1} due to the stretching of the amide carbonyl (CO) bond, and two absorption bands appeared at the frequency (1608, 1462) cm^{-1} due to the stretching of the aromatic (C=C) bond, and there was an absorption band at The frequency (1384) cm^{-1} is related to the (CO) bonding, and an absorption beam appeared at the frequency (1257) cm^{-1}, which was due to the stretching of the (CN) bond, and an absorption beam appeared at the frequency (1180) cm^{-1}, which was due to the stretching of the (N-N) bond. As shown in figure (5), these packages were close to what is found in the literature [26, 27].

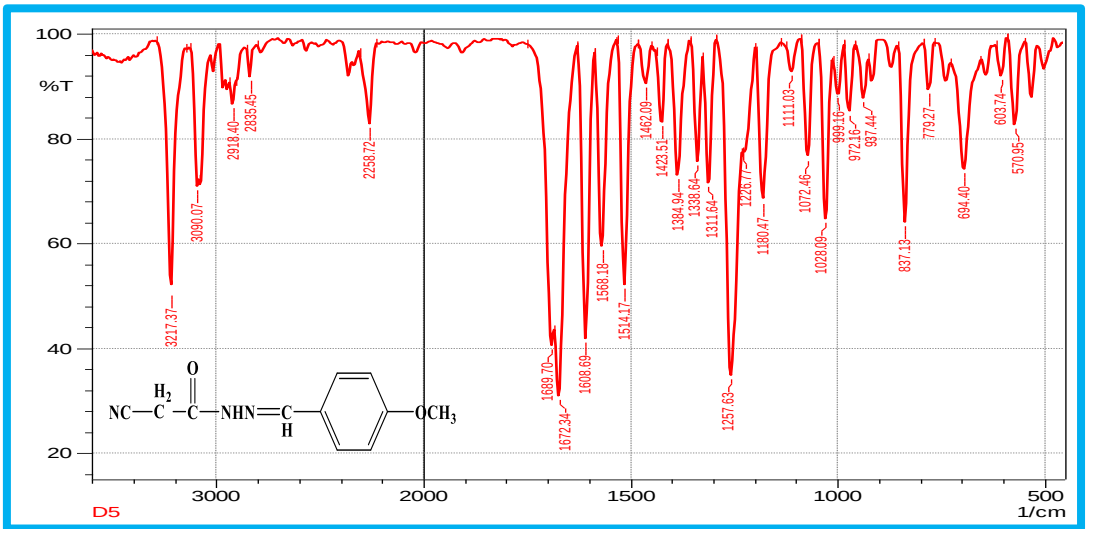


Figure 5: FTIR spectrum of D5

The nuclear magnetic resonance spectrum of proton for compound [D5] using a solvent (DMSO-d^6), showed that a signal appeared at the position (7.99) ppm attributed to the proton of the (NH) group, and the appearance of a single signal at the position (7.84) ppm that returned to the proton of the azomethine group (HC=N), as well as the appearance of a multiple signal at the site (6.88-7.56) ppm attributed to the aromatic ring protons, and the appearance of a single signal at the site (4.08) ppm attributed to the proton of the (CH_2) group, and the appearance of a single signal at the site (4.08) ppm attributed to the proton of the (CH_2) group. A single signal in the position (3.68) ppm attributed to the protons of the (CH_3) group, as well as the appearance of a signal in the position (3.25) ppm attributed to water protons, and the appearance of a signal in the position (2.39, 2.40, 2.41) ppm attributed to solvent protons (DMSO-d^6) [28], as in figure (6).

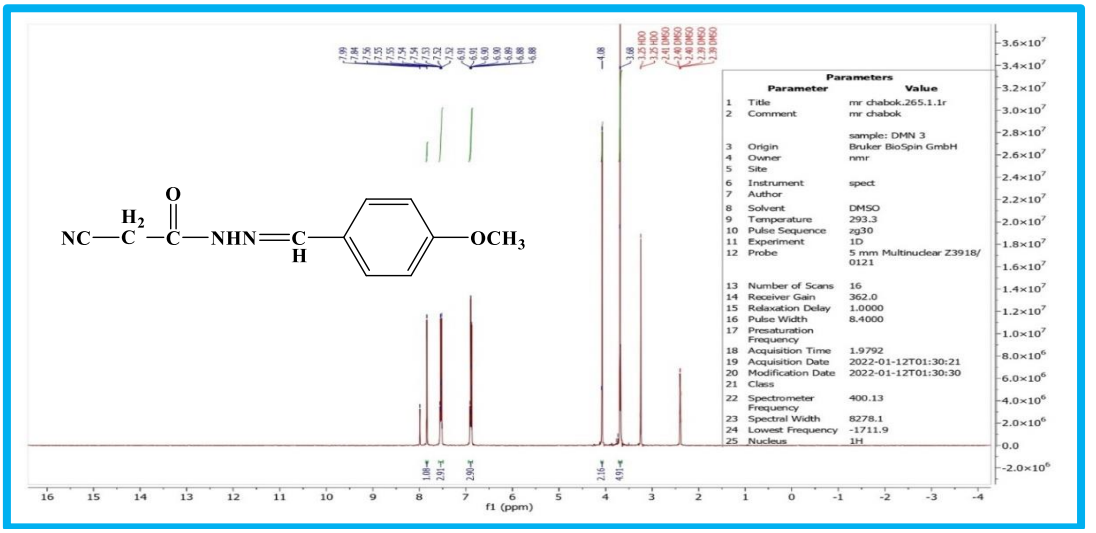


Figure 6: ^{1}H-NMR spectrum of D5

The nuclear magnetic resonance spectrum of carbon for compound [D5] using a solvent (DMSO-d^6), showed that a signal appeared at the site (24.73) ppm attributed to the carbon of the (CH_2) group, and the appearance of a signal at the site (55.75 ppm attributed to (CH_3) group carbon, and the appearance of signals at the position (114.73-129.36) ppm belonging to the aromatic benzene ring carbons, as well as the appearance of a signal at the site (159.08) ppm attributed to the carbon group (CN), and the appearance of a signal at the site (161.34) ppm attributed to the carbon of the group (C=N), the appearance of a signal at the site (164.98) ppm attributed to the carbon of carbonyl group (CO), and the appearance of signals at the site (39.35-40.60) ppm attributed to the solvent carbon (DMSO-d^6) [29], as in figure (7).

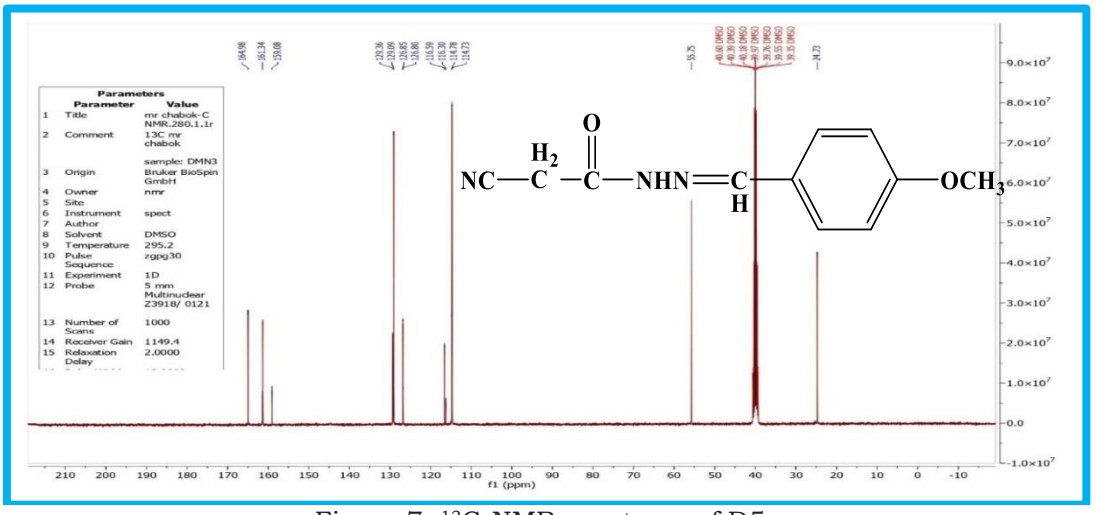
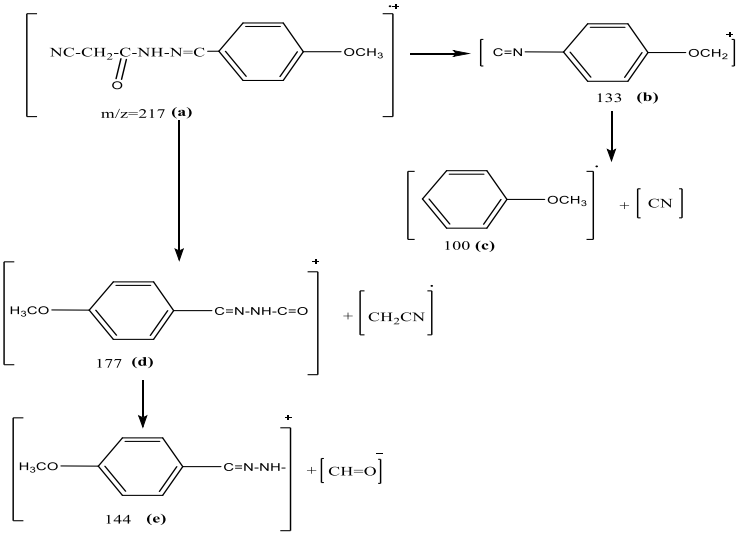


Figure 7: ^{13}C-NMR spectrum of D5

The mass spectrum of compound (D5), according to the (figure 8), showed a base peak at (133) that belongs to the group [b]. A peak appeared at (100) that corresponds to the group [c]. A peak at (217) is attributed to the molecular weight of the compound molecule (a) in addition to the appearance of a peak at (177) for group [d] and a peak at (144) for group [e][30].



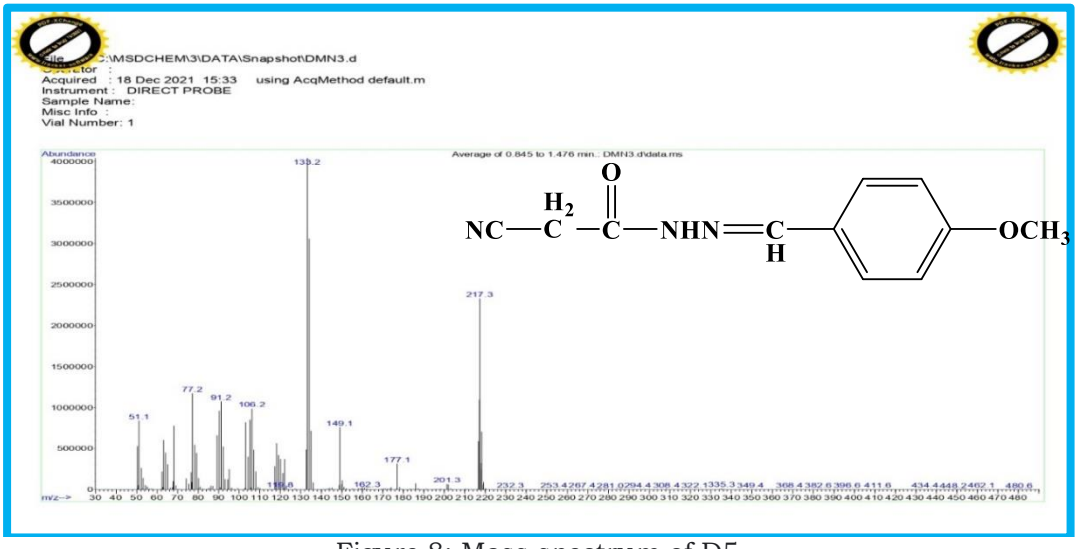


Figure 8: Mass spectrum of D5

The synthesis of Oxazepine compounds from reaction (Schiff bases with phthalic anhydride), the FTIR spectra of compound D_{10} showed that the band of azomethine group (C=N) disappeared, and absorption bands appeared at frequency (3215) cm^{-1} due to the stretching of the (NH) bond, and the absorption band appeared at frequency (3090) cm^{-1} due to the stretching of the (CH) aromatic bond, as well as the appearance of two absorption bands at frequency (2918, 2835) cm^{-1} due to the stretching of the (CH_3) bond, in addition to the appearance of an absorption band at frequency (2256) cm^{-1} due to the stretching of the (CN) bond), and it was observed that a new absorption band appeared at the frequency (1734) cm^{-1} due to the stretching of the lactone bond, And the appearance of an absorption band at frequency (1691) cm^{-1} due to the stretching of the lactam bond, and the appearance of an absorption band at frequency

(1672) cm^{-1} due to the stretching of the amide carbonyl bond (CO), and two absorption bands appeared at frequency (1608, 1514). cm^{-1} is due to the stretching of the aromatic (C=C) bond, and an absorption band appeared at the frequency (1338) cm^{-1} that was due to the stretching of the (CO) bond, and an absorption band appeared at the frequency (1257) cm^{-1} that was related to the stretching of the (CN) bond, and it appeared An absorption beam at a frequency of (1180) cm^{-1} is due to the stretching of (N-N) bond, as shown in (figure 9), and these beams were close to what is found in the literature [31].

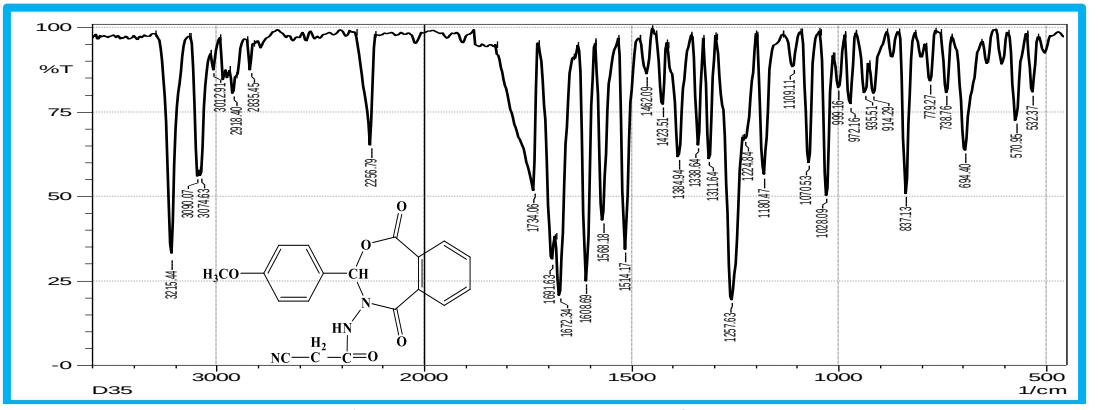


Figure 9: FTIR spectrum of D10

The nuclear magnetic resonance spectrum of proton for compound [D10] using a solvent (DMSO-d^6), showed that a signal appeared at the position (7.99) ppm attributed to the proton of the (NH) group, and the appearance of a multiple signal at the position (6.91-7.84) ppm. The appearance of a single signal at the position (6.88) ppm attributed to the proton of the (CH) group of the seven-membered ring, the appearance of a single signal at the position (4.08) ppm attributed to the proton of (CH_2) group, and the appearance of a single signal at the position (4.08) ppm attributed to the proton of (CH) group. A single signal at the position (3.69) ppm attributed to the protons of (CH_3) group, as well as the appearance of a signal at the position (3.27) ppm attributed to the water protons, and the appearance of a signal at the site (2.39, 2.40, 2.41) ppm attributed to solvent protons (DMSO-d^6) [33], as in figure (10).

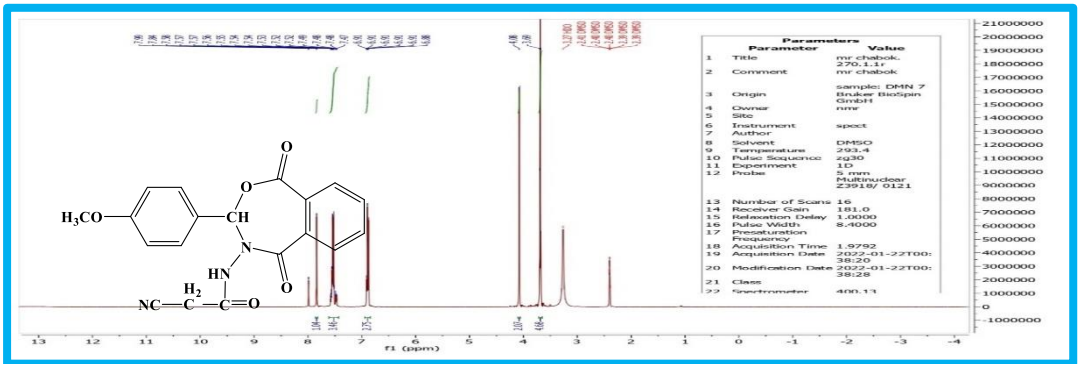


Figure 10: ^{1}H-NMR spectrum of D10

The nuclear magnetic resonance spectrum of carbon for compound [D10] using a solvent (DMSO-d^6), showed that a signal appeared at the site (24.25 ppm) attributed to the carbon of the (CH_2) group, and a signal appeared at the site (55.76) ppm attributed to (CH_3) group carbon, and the appearance of a signal at the position (95.10) ppm attributed to the carbon of the (CH) group of the heptathlon, and the appearance of signals at the position (114.74-148.17) ppm attributed to the aromatic benzene ring carbons, as well as the appearance of a signal at the site (159.38) ppm attributed to the carbon of the (CN) group, and a signal appeared at the site (161.32) ppm attributed to the carbonyl group carbon (CO), and a signal appeared at the site (164.80) ppm attributed to the carbon of the lactam group (CO), And the appearance of a signal in the position (169.22) ppm attributed to the carbon of the lactone group (CO), and the appearance of the signals in the position (39.34-40.59) ppm attributed to the carbonate of the solvent (DMSO-d^6), as in (figure 11).

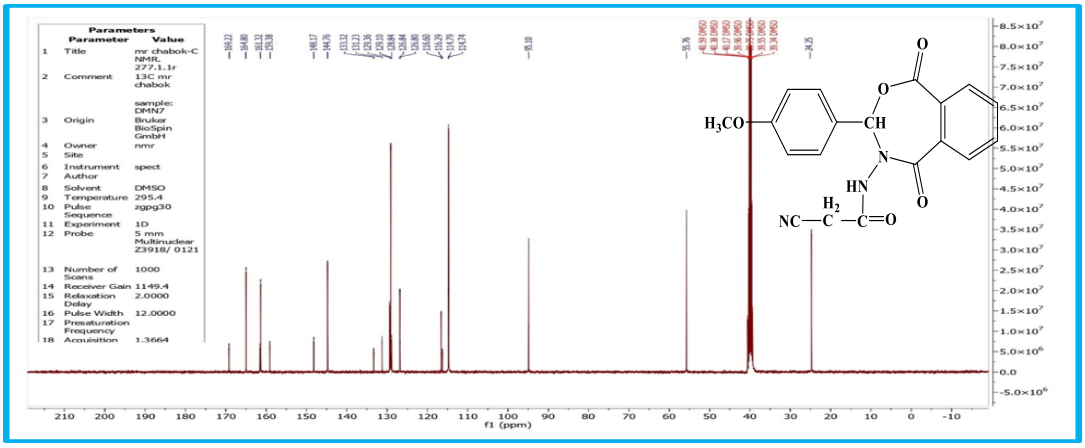


Figure 11: ^{13}C-NMR spectrum of D10

3.1. Evaluation of antibacterial activity

Using the agar diffusion technique[18] , all of the produced compounds were evaluated for antibacterial activity against gram-negative bacteria (Escherichia coli) and gram-positive bacteria (Staph. aureus). To achieve a concentration of 1 ppm, each chemical was dissolved in DMSO. After that, the plates were incubated at 37°C for 24 hours before being analyzed. The inhibitory zones that developed were measured in millimeters. For testing the potential actions of these compounds, conventional medicines such as gentamycin were utilized. All substances were evaluated at various doses (0.001 mg/mL), with DMSO serving as both a solvent and a control. The antibacterial activity was measured using the inhibition zone diameter in millimeters (IZD). The minimum inhibitory concentration (MIC, g/mL), defined as the lowest concentration necessary to stop bacteria from growing, was measured for all substances compared to the control. Antibacterial screening results indicated that (D_{2-6}) and (D_{2-6}) were the most common (D_{7-11}). Compounds (D_{2-6}) have strong antibacterial action against gram-positive and gram-negative bacteria (Escherichia coli). In contrast to Gentamycin derivatives, compounds (D7-11) showed strong antibacterial action against gram-positive bacteria (Staph. aureus) and showed high activity against all

microorganisms tested.Compounds with the highest activity (MIC= 22.8 g/mL) were identified. showed strong antibacterial action against gram-negative bacteria (Escherichia coli) and showed high activity against all microorganisms tested Compounds with the highest activity (MIC= 22.6 g/mL) were identified. The outcomes are shown in figures (12 and 13).

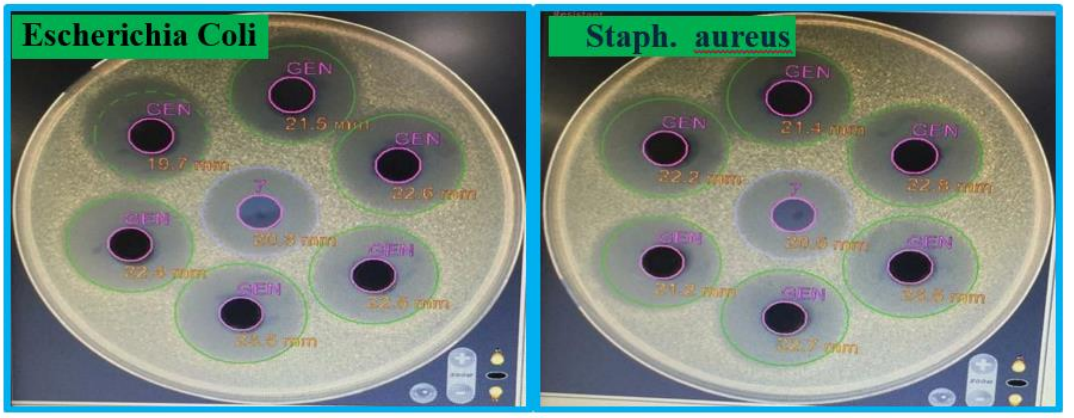


Figure 12: Compound (D5) inhibits the growth of bacteria Escherichia Coli and compound (D10) inhibits the growth of bacteria Staph. Aureus

4. Conclusions

The spectroscopic and physical measurements demonstrated correctness of structure of the synthesized compounds. The antibacterial study of (D_{2-6}) and (D_{7-11}) compounds showed antibacterial activity against the two types of bacteria (E. coli and Staph. aureus) under study at the low concentrations of (0.001 mg/mL) compared with the antibiotic Gentamycin.

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