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Article

Synthesis of a Series of Hexacyclic Compounds, Including the Pyran Ring Derived from Chalcone, and Evaluation of Their Biological Effectiveness

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Abstract: This research aimed to synthesize a series of hexacyclic heterocyclic compounds, including pyran ring derivatives from chalcone, and to evaluate their biological effectiveness. The synthesis involved the reaction of chalcone derivatives with alpha-cyano ethyl acetate and malononitrile in a basic medium using sodium hydroxide. The resulting compounds were characterized by infrared (IR) spectroscopy, proton nuclear magnetic resonance (H-NMR), and carbon-13 nuclear magnetic resonance (13C-NMR). The biological activity of the synthesized compounds was tested against various bacteria, showing significant inhibitory effects compared to standard antibiotics. The results suggest that these compounds hold potential as therapeutic agents, warranting further investigation into their biological pathways and effects.

Keywords: Hexacyclic Compound Synthesis, Pyran Ring, Antimicrobial Biological Activity, Alpha-Beta Unsaturated Chalcone and IR, H-NMR, 13C-NMR Spectral Characterization

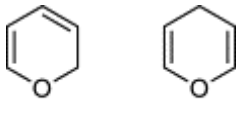
Introduction

Heterocyclic

Compounds an organic compound containing one carbon atom as a minimum associated with a different atom and oxygen, nitrogen, or sulfur is one of the elements with a high ability to form heterocyclic compounds, because the angle of valence of these elements is very close to the angle of valence of the carbon element, so it can replace one or more atoms in an aromatic or aliphatic ring system similar to the benzene ring system (C_6H_6) [1,2]. This characterization reveals that almost 70% of pharmacological and pharmaceutical compounds contain at least one episode of heterogeneous rings, as do numerous natural compounds that fit the same description and contain heterocyclic rings" [3,4].

Pyaran

Although the simple, unsaturated pyran ring systems seen in 2H-pyran 1 and 4H-pyran 2 are unstable and cannot be synthesized by biosynthetic pathways, six-membered oxygen heterocyclic ring derivatives are found in a wide range of naturally occurring compounds [5,6].



1 **2**

Pyran derivatives are a useful class of heterocyclic compounds that are widely distributed in nature [7], and pyran and molten pyran derivatives have received a lot of attention because of their association with various types of biological properties, such as benzo(b) pyrene derivatives, which have been found to exhibit anti-cancer activities against three lines of human cells even at very low concentrations [8]. 2-amino-4H-pyraan is used as a photoactive dye and biodegradable agricultural chemical [9].

Materials and Methods

Chemicals used: Chemicals prepared by Aldrich, BDH Thomas, Fluka, and Merck were used.

Preparation of Pyran Ring Derivatives (B35-42) [10]:

In a circular flask of size (100 ml), dissolve (0.01 ml) of the prepared Chalcone derivatives [B1-10] in (10 ml) of ethanol and then add to it a solution of (0.01) alpha-cyano-ethyl acetate in (10 ml) of ethanol and then add to it 10 ml of (10%) sodium hydroxide and rise the mixture for 6 hours with constant stirring, cool the solution and add to the ice crushed and equivalent by adding drops of hydrochloric acid Filtration separates the concentrate from the precipitate, which is then washed with cold water and recrystallized from methanol; Table (1) shows the physical parameters.

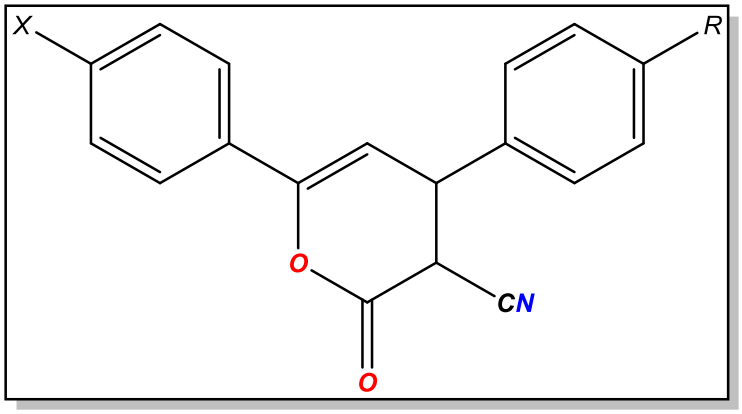


Table 1. Some Physical Characteristics and Percentages of Compounds (B1-8)

Comp. No.	X	M.P. (˚C)	Yield (%)	Color	Formula
B_1	4-Br	160-163	86	Green	$C_{18}H_{11}NO_2Br_2$
B_2	4-Cl	170-171	94	White	$C_{18}H_{11}NO_2BrCl$
B_3	4-CH3	131-132	96	Yellow	$C_{19}H_{14}NO_2Br$
B_4	4-OCH3	182-183	34	Yellow	$C_{19}H_{14}NO_3Br$
B_5	4-Br	189-192	66	Orange	$C_{19}H_{14}NO_2Br$

B_6	**4-Cl**	**96-97**	**41**	**Yellow**	$C_{19}H_{14}NO_2Cl$
B_7	**4-CH3**	**112-113**	**62**	**Brown**	$C_{20}H_{17}NO_2$
B_8	**4-OCH3**	**153-154**	**53**	**Brown**	$C_{20}H_{17}NO_3$

Preparation of derivatives of episode 2 - amino Pyran (B1-8) [10]:

In a circular flask volume of (100) ml, dissolve (0.01 ml) of the prepared chalcone derivatives (B1-10) in (10 ml) of ethanol, then add to it a solution (0.01 ml) of malononitrile dissolved in 10 ml of ethanol, and then add to it (10 ml) of a solution (10%) sodium hydroxide and rise the mixture for 6 hours continuous stirring, then concentrate the solvent and cool the mixture and add to the ice crushed, then add drops of concentrated hydrochloric acid to equalize the mix Then it is filtered, washed with cold water, and recrystallized in methanol. Table 2 shows the physical properties and percentages of derivatives.-2-aminopyran (B1-8).

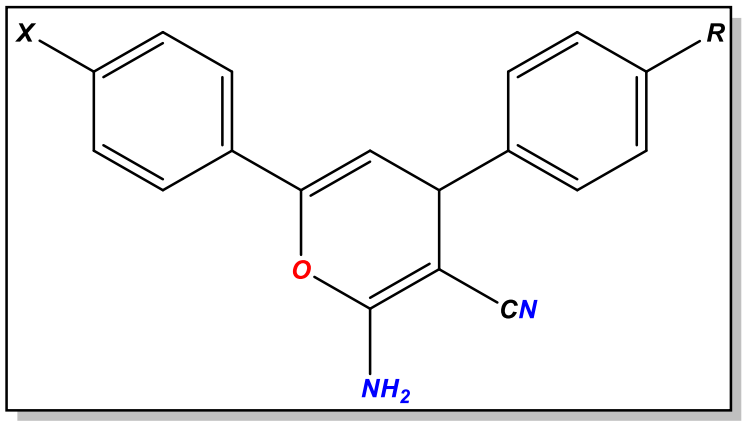


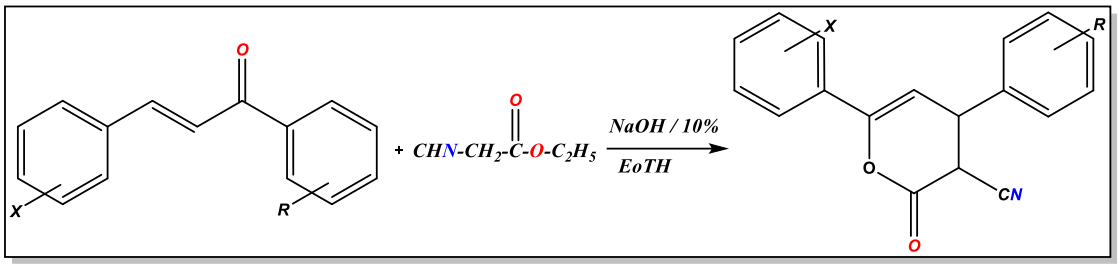
Table 2. Some Physical Constants and Percentage of Compounds (B1-8)

Comp. No.	X	M.P. (˚C)	Yield (%)	Color	Molecular Formula
B_1	4-Br	180-183	58	Blue	$C_{18}H_{12}N_2Br_2$
B_2	4-Cl	167-170	93	Brown	$C_{18}H_{12}N_2BrCl$
B_3	4-CH3	170-171	51	Yellow	$C_{19}H_{15}N_2Br$
B_4	4-OCH3	159-161	96	White	$C_{19}H_{15}N_2OBr$
B_5	4-Br	157-159	95	Yellow	$C_{18}H_{12}N_2Br_2$
B_6	4-Cl	130-132	79	Yellow	$C_{19}H_{15}N_2Cl$
B_7	4-CH3	93-96	43	White	$C_{20}H_{18}N_2$
B_8	4-OCH3	73-74	51	Yellow	$C_{20}H_{18}N_2O$

Result and Discussion

Diagnosis of 2-Oxo-Pyran compounds (B35-42):

Through the interaction of galuconate with alpha-cyano ethyl acetate in a basic medium, the babaran was obtained as shown in the following equation:



The validity of the compounds prepared by spectral methods was confirmed, with the infrared spectrum (FT-IR) showing a beam at the frequency (3024-3338cm^{-1}) belonging to the expansion group (Ar-H), a beam at the frequency (2142-2364cm^{-1}) belonging to the expansion of the group (CN), a beam at the frequency (1577-1679cm^{-1}) belonging to the expansion of the group (C=O), and a beam at the frequency (1209-1298cm^{-1}) belonging to the expansion of the group (C-O) [11].

Table 3. Compounds Infrared Absorption Spectrum Packets (B1-8)

Comp. No.	R	FT.I.R υ cm^{-1}(KBr)				Others
		Ar-H	C≡N	C=O	C-O	
B1	4-Br	3056	2221	1587	1271	C-Br(567)
B2	4-Cl	3058	2312	1681	1321	C-Br(535) C-Cl(694)
B3	4-CH_3	3178	2227	1643	1218	C-Br(534)
B4	4-OCH3	3024	2333	1665	1259	C-Br(582)
B5	4-Br	3047	2195	1676	1251	C-Br(549) C-Cl(752)
B6	4-Cl	3338	2362	1679	1265	C-Br(584) C-O(1255)
B7	4-CH_3	3115	2190	1679	1298	CH3(2881)
B8	4-OCH_3	3221	2364	1677	1249	CH3(2921)

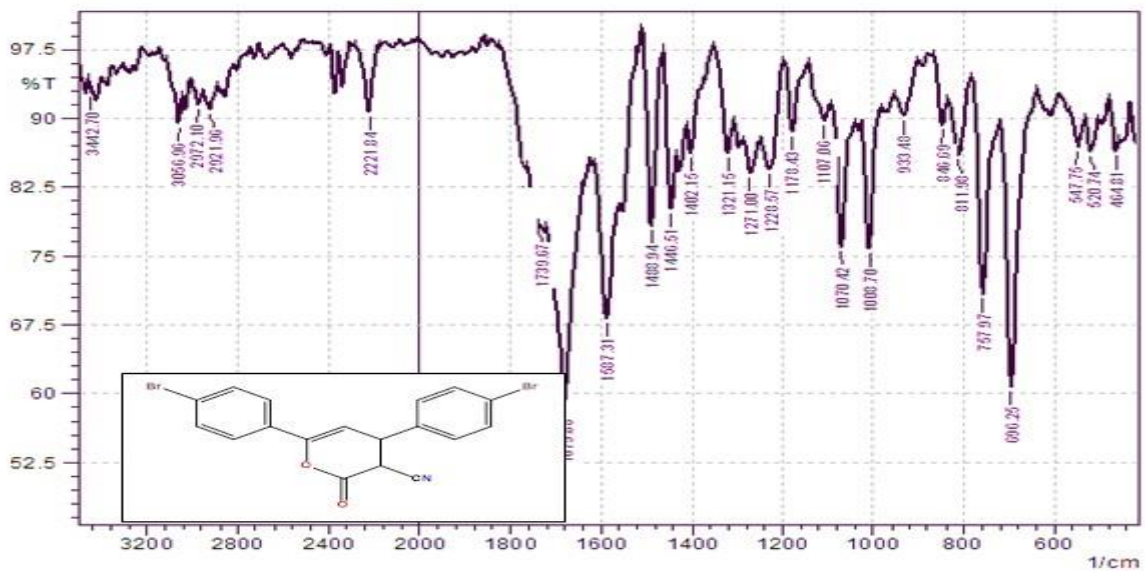
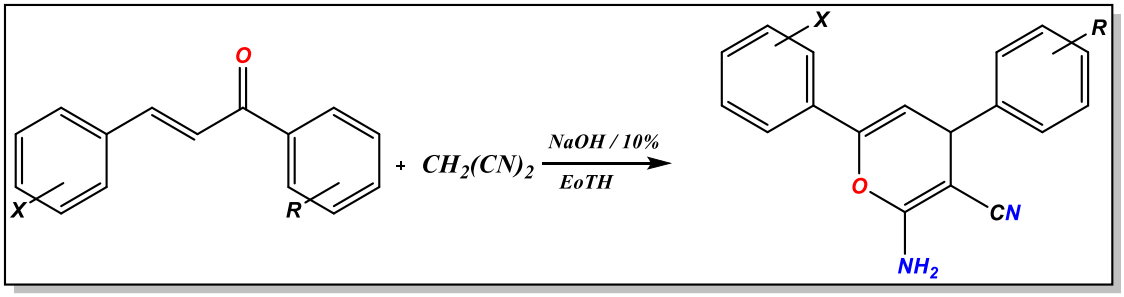


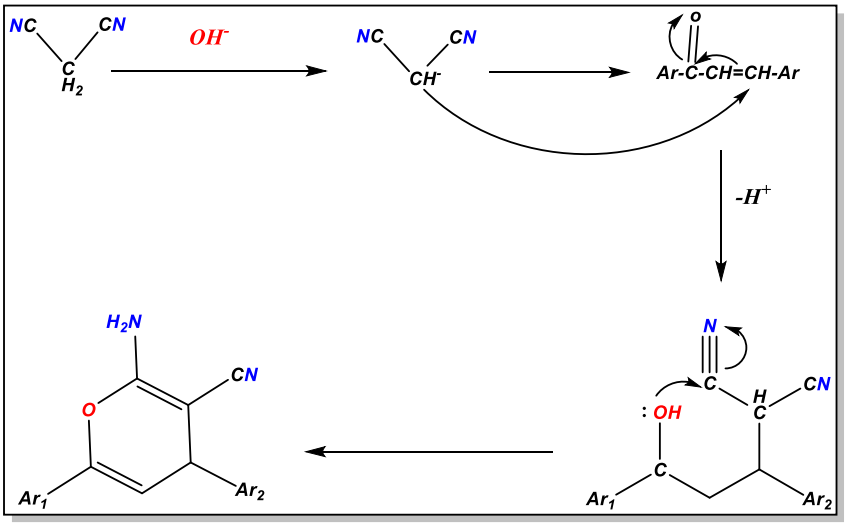
Figure 1. Composite Infrared Spectrum (B1)

Diagnosis of 2-aminopyran compounds (B1-8):

Through the interaction of chalcones with malononitrile in a base medium, (2-aminopyrane) was obtained as in the following equation:



Proposed Mechanical:



Spectral methods confirmed the structures' validity, revealing a beam at frequency (3356-3450cm^{-1}) belonging to the stretch group (NH_2), a beam at frequency (3035-3197cm^{-1}) belonging to the

stretch group (Ar-H), a beam at frequency (2185-2223cm^{-1}) belonging to the group (CN), and a beam at frequency (1205-1298cm^{-1}) belonging to the stretch group" (C-O) [12].

Table 4. Compounds Infrared Absorption Spectrophotometer Packs (B1-8)

Comp. No.	R	FT.I.R υ cm^{-1}(KBr)				Others
		NH	Ar-H	C≡N	C-O	
B1	4-Br	3440	3076	2221	1205	C-Br(516)
B2	4-Cl	3446	3035	2185	1298	C-Br(545) C-Cl(694)
B3	4-CH_3	3477	3068	2198	1261	C-Br(549)
B4	4-OCH3	3356	3040	2223	1251	C-Br(554)
B5	4-Br	3375	3062	2208	1273	C-Br(565) C-Cl(759)
B6	4-Cl	3440	3035	2223	1255	C-Br(
B7	4-CH_3	3375	3182	2212	1249	C-Br(584)
B8	4-OCH_3	3450	3197	2217	1245	CH_3(2989)

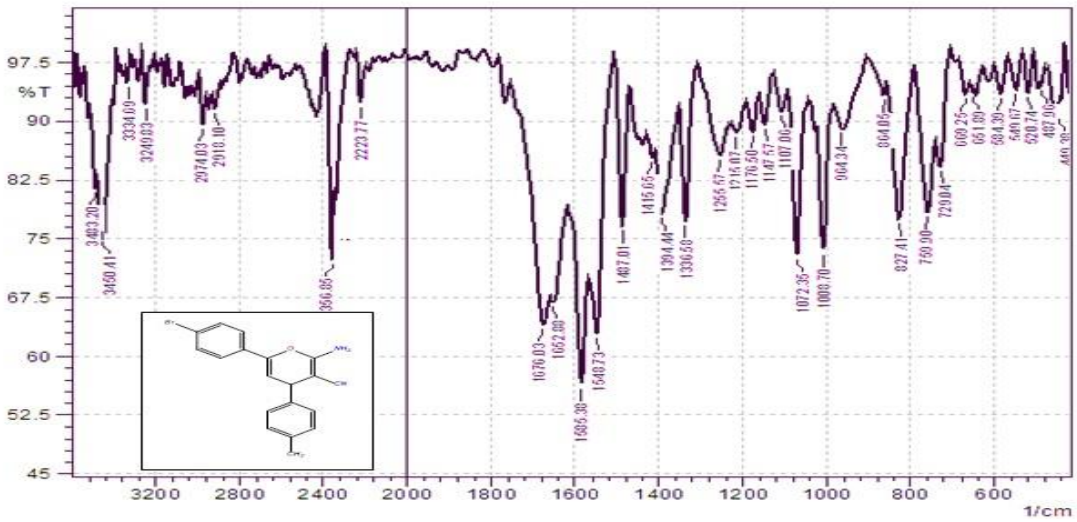
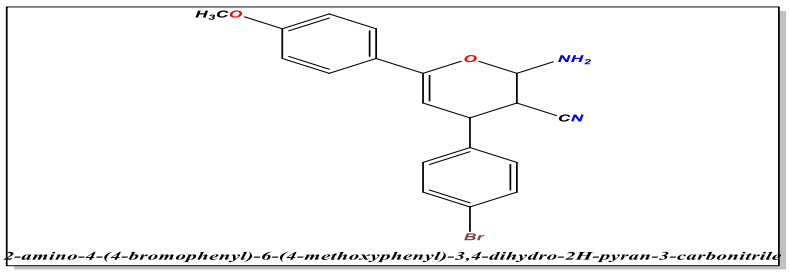


Figure 2. Composite Infrared Spectrum (B8)

Proton Nuclear Magnetic Resonance Resonance Spectrophotometer (H-NMR) for B1:

B1 was diagnosed by the 1H-NMR spectrum using DMSO-d6 as a solvent and showed a signal at the site (δ:2.6ppm) belonging to the solvent used (DMSO-d6), the appearance of a signal at the range (δ:3.9 ppm) attributed to a proton (2H/O-CH3), the appearance of a signal within the range (δ:3.2-5.3 ppm) belonging to the protons of the pyran ring (3H/Ar-CH) and the appearance of multiple signals within the range (δ; 6.7-8.1ppm) attributed to the protons of the aromatic ring (2H/Ar-CH) and the appearance of a signal at the range (δ:8.4 ppm) returning to the protons of the amine group" (2H/C-NH2) [13].



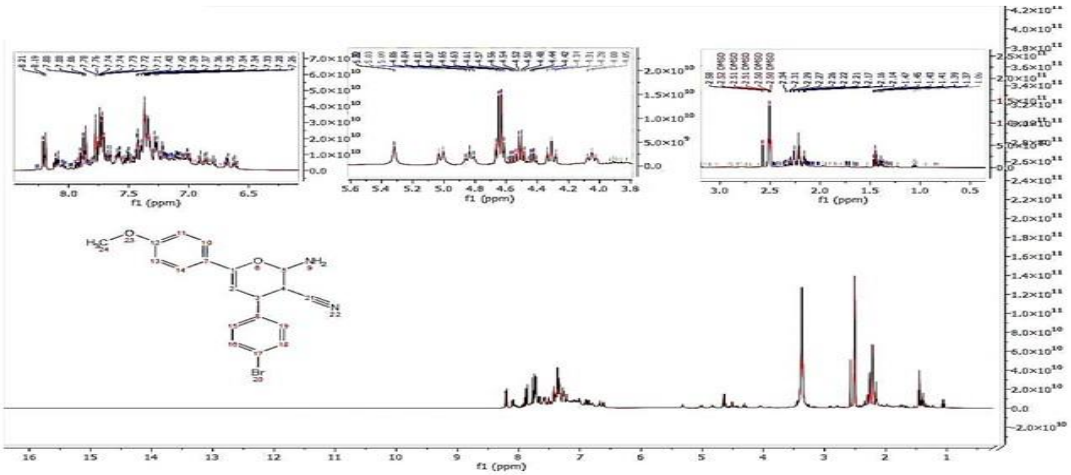
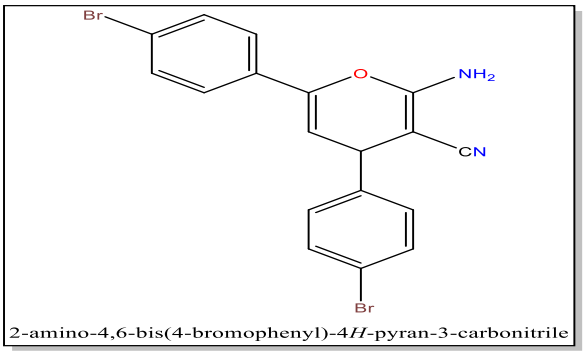


Figure 3. The nuclear magnetic resonance spectrum (H-NMR) of B4

Proton Nuclear Magnetic Resonance Spectrum (H-NMR) for B3:

B3 was diagnosed by the 1H-NMR spectrum using DMSO-d6 as a solvent and showed a signal at the site (δ:1.6 ppm) belonging to the protons of the proverbial group (H/CH3), showed a signal at the site (δ:2.6ppm) belonging to the solvent used (DMSO-d6), the appearance of a signal at the range (δ:3.4-3.7 -4.8ppm) attributed to the protons of the pyran ring (2H/Ar-CH), the appearance of a signal at the range (δ:3.9 ppm) belonging to the proton (2H/O-CH3) and the appearance of multiple signals within the range (δ; 6.9-7.9ppm) attributed to aromatic ring protons (2H/Ar-CH) and the appearance of a signal at the range (δ:8.3 ppm) returning to the protons of the amine group (2H/C-NH2) [14].



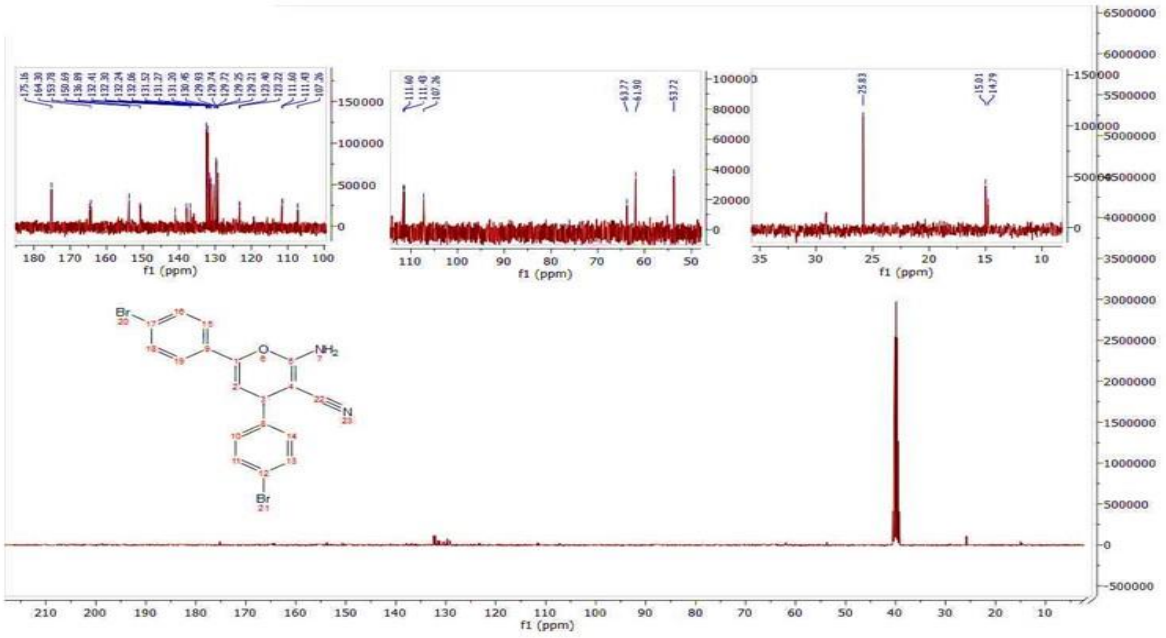
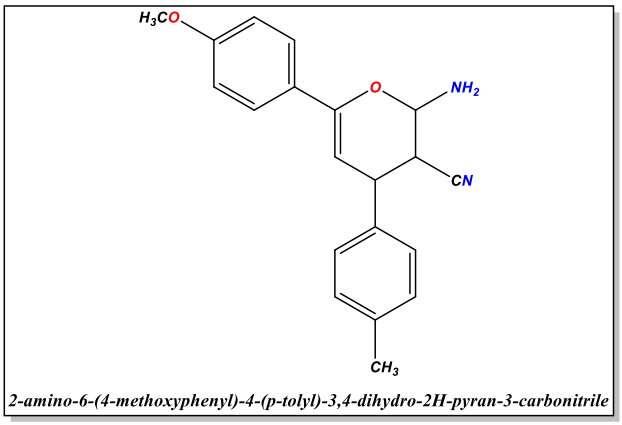


Figure 4. The nuclear magnetic resonance spectrum (13C-NMR) of B3

^{13}C. NMR spectrum of B1:

The compound (B1) was studied by magnetic resonance spectrophotometer (13C-NMR) where it showed a signal at frequency (δ:14 ppm) belonging to the carbon atoms of the pyran ring (C-C) and the appearance of a signal at the frequency (δ:27 ppm) belonging to the carbon atom of the proverb group (C-CH_3) and the appearance of a signal at the frequency (δ:40 ppm) The solvent used (DMSO-d6) and the appearance and signal at the frequency (δ:114 ppm) belonging to the carbon atom group (O-CH_3) The appearance of a signal at the ejection (δ:124 ppm) Returning to the amino carbon atom (C-NH_2) and the appearance of A signal at the frequency (δ:126 ppm) belongs to the carbon atom of the pyran ring (C = C) and the appearance of a signal at the frequency (δ:128 ppm) returns to the carbon atom of the pyran (C = C) and the appearance of a signal at the frequency (δ:129 ppm) returns to the aromatic ring carbon atom (C = C) and the appearance of a signal at the frequency (δ:130 ppm) returns to the aromatic ring carbon atom (C-C) and the appearance of a signal at the frequency (δ:130 ppm) returns to the carbon atom of the cyanide group ($C \equiv N$) and the appearance of a signal when the response (δ: 131ppm) returns to the aromatic ring carbon atom (C=C) and the appearance of a signal at the frequency (δ: 132 ppm) returns to the aromatate ring carbon atomAromatic (C-C) and the appearance of a signal at the frequency (δ:135 ppm) due to the carbon of the aromatic ring (C = C) and the appearance of a signal at the frequency (δ; 136 ppm) belonging to the carbon atom of the aromatic ring (C-C) and the appearance of a signal at the frequency (δ:136ppm) returns to the carbon atom of the aromatic ring (C-C) and the appearance of a signal at the frequency (δ:156 ppm) belongs to the carbon atom of the aromatic ring (C-C) and the appearance of a signal at the frequency (δ:157 ppm) returns to the carbon group (C-O) in the byron ring and the appearance of a signal At the frequency (δ; 167 ppm) it returns to the C-O group carbon atom in the aromatic ring [15].



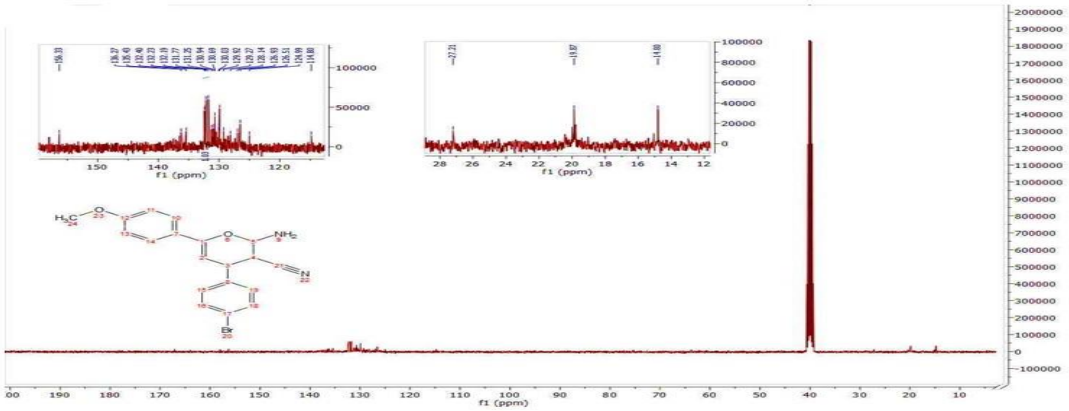
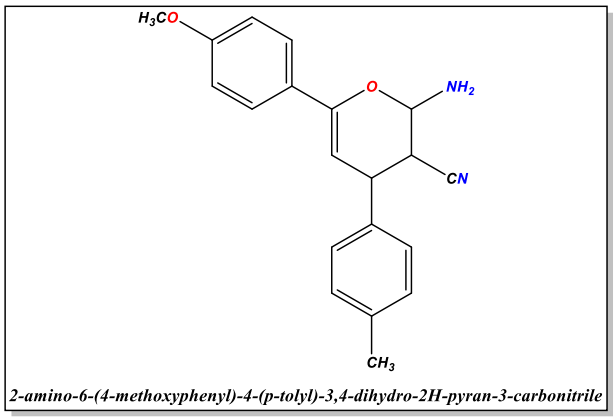


Figure 5. The NMR spectrum of 1

13C- NMR Spectrophotometer (B3)

The compound (B3) was studied by magnetic resonance spectrophotometer (13C-NMR) where it showed a signal at frequency (δ:15 ppm) belonging to the carbon atoms of the proverbial group (C-CH3) and the appearance of a signal at the frequency (δ:28 ppm) belonging to the carbon atom of the pyran ring (C-C) and the appearance of a signal at the frequency (δ:40 ppm) The solvent used (DMSO-d6) and the appearance and signal at the frequency (δ:55 ppm) belonging to the carbon atom group (O-CH3) The appearance of a signal at the ejection (δ:71 ppm) belonging to the carbon amino atom (C-NH2) and the emergence of A signal at the frequency (δ:92 ppm) returns to the carbon atom of the pyran ring (C=C), the appearance of a signal at the frequency (δ:113 ppm) returns to the carbon atom of the pyran (C=C), the appearance of a signal at the frequency (δ:114 ppm) returns to the aromatic ring carbon atom (C-C) and the appearance of a signal at the frequency (δ:116 ppm) Returns to the carbon atom of the cyanide group ($C \equiv N$) and the appearance of a signal at the frequency (δ: 124ppm) It returns to the aromatic ring carbon atom (C=C) and the appearance of a signal at the frequency (δ: 127 ppm) It returns to the aromatic ring carbon atom (C-C) and the appearance of a signal at the frequency (δ:129 ppm) It returns to the aromatic ring carbon atom (C = C) and the appearance of a signal at the frequency (δ; 132 ppm) It returns to the aromatic ring carbon atom (C = C) and the appearance of a signal at the frequency (δ:136ppm) Returns to the ring carbon atom Aromatization (C-C) and the appearance of a signal at the frequency (δ:146 ppm) belonging to the carbon atom of the aromatic ring (C-C), the appearance of a signal at the frequency (δ:157 ppm) belonging to the carbon group (C-O) in the byron ring and the appearance of a signal at the frequency (δ; 198 ppm) belonging to the carbon atom group (C-O) in the aromatic ring [16].



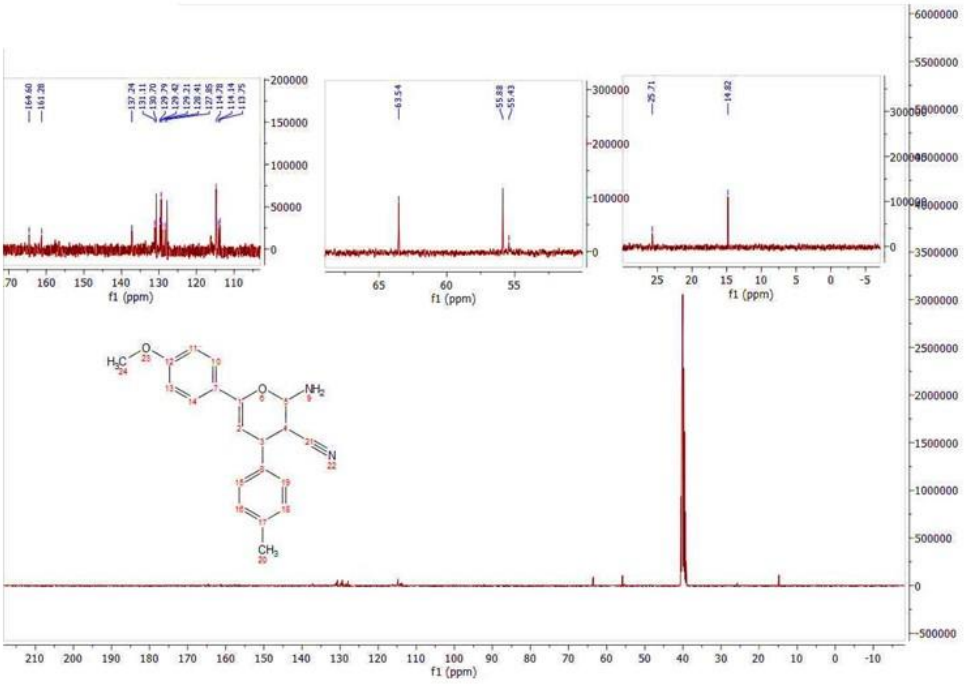


Figure 6. The nuclear magnetic resonance spectrum (13C-NMR) of B3

Biological activity of some prepared compounds

The study of the biological efficacy of compounds prepared in certain concentrations showed that most of these compounds have an antidote to the types of bacteria studied compared to the antibiotic (Ciprofloxacine), which is a broadly classified antibiotic, especially these two types of bacteria studied, in addition to many types, and it also has a large inhibitory diameter as it gives high selectivity when studying the sensitivity of bacteria to the prepared compounds, and since this antidote is used to treat many infections and diseases such as urinary tract infections, especially those that occur as a result of infection with bacteria Colon and Staphylococcus aureus bacteria [17-20], treats simple cystitis in females caused by colon bacteria, and treats chronic bacterial prostatitis caused by colon bacteria and Staphylococcus aureus, in addition to infections of the lower respiratory tract, sinusitis, arthritis and bones, and is also used to treat diarrhea caused by infection with colon bacteria, and is also effective in treating typhoid, so the compounds prepared in this study [21-23], numbering (10) compounds used on different types of chromium-negative bacteria, have recorded a high antibody efficacy against the studied bacteria, and compared to the antibiotic mentioned, it is possible to use these compounds as a treatment for infections and the same pathological conditions mentioned above after investigating the vital path of these compounds, their side effects and the amount of their accumulation in animal tissues [24-27], and the results shown in Table (5) indicate that most of the prepared compounds have the

ability to Inhibition of bacteria used by different concentrations of compounds (0.01, 0.001, 0.0001mg/ml) as the inhibition diameter ranges between (10 ml) minimum inhibition diameter to (20) ml maximum inhibition diameter size the following table shows the inhibitory effectiveness of a number of prepared compounds [28-30]:

Table 5. Inhibitory Activity of Some Compounds Prepared for the Growth of a Number of Negative and Positive Bacteria

Com No	Conc. mg/m	*S.A*	*E.F*	*P.P*	*K.P*	*f*
B1-27	0.01	/	14	/	/	18-cn10
	0.001	/	10	/	/	/
	0.0001	/	12	/	/	/
B3-30	0.01	/	10	/	/	20-cn10
	0.001	/	8	8	/	/
	0.0001	/	12	/	/	/
B5-37	0.01	/	/	/	/	8-cn10
	0.001	/	/	/	/	/
	0.0001	/	/	/	/	/

Figure 7. Illustrates the effect of inhibiting the growth of Enterococcus faecal bacteria

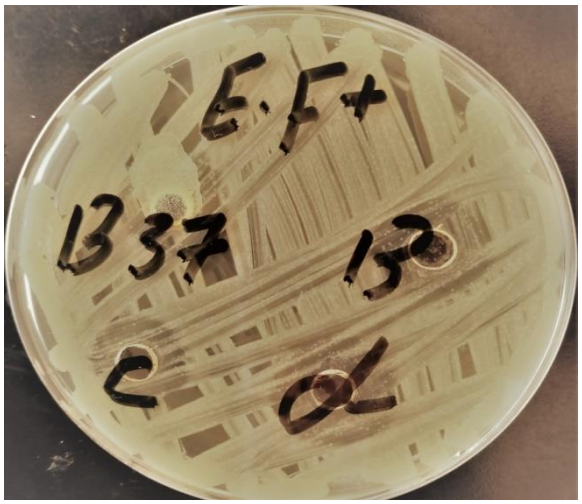


Figure 8. Illustrates the effect of inhibiting the growth of Enterococcus faecalia bacteria

Figure 9. The effect of inhibiting the growth of Pseudomonas putida bacteria

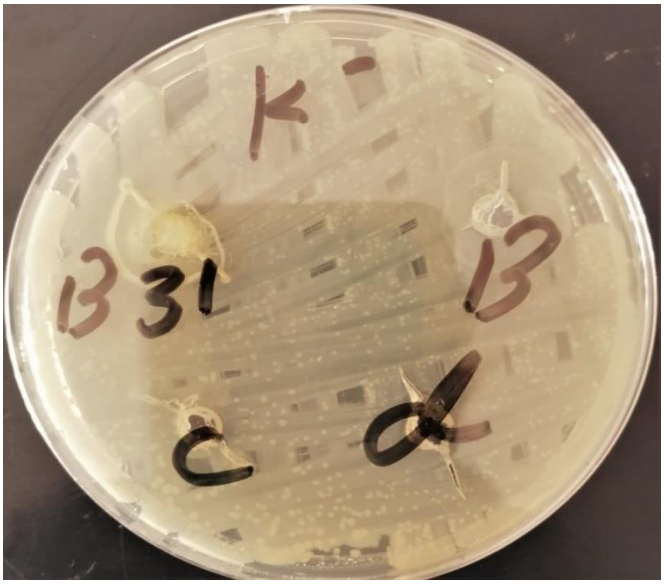


Figure 10. Illustrates the effect of inhibiting the growth of Klebsiella pneumonia bacteria

Conclusion

Spectroscopic and physical measurements showed the health of the prepared compounds. The compounds showed high purity. The compounds showed good product yield. The compounds showed good biological activity against the bacteria used".

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