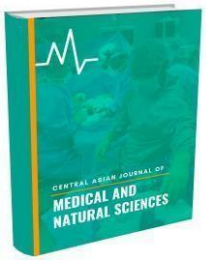

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Article

Preparation And Evaluation Of The Biological Activity Of A 2-Amino Pyran Ring Using A Solid Base Catalyst

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Abstract: The research included preparing a basic catalyst by reacting potassium nitrate with alumina to produce the heterogeneous solid basic catalyst and then introducing the catalyst as a catalyst in preparing the 2-amino pyrane ring by reacting the prepared chocolate with malononitrile and using only stirring without sublimation or heating. The compositions were validated using physical measurements such as percentage, melting point, and colour and spectroscopic measurements such as proton and carbon nuclear magnetic resonance and infrared spectra. The nanoscale size of the catalyst was also confirmed by scanning electron microscope (SEM) analysis. Two types of positive bacteria were used—gram and gram-negative to verify biological activity.

Keywords: catalyst, pyran, biological activity.

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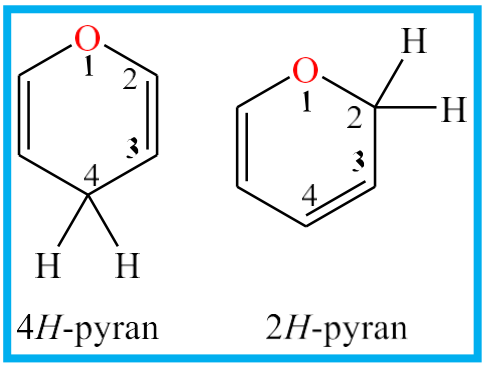
1. Introduction

Catalyst The term catalyst was originally defined by the scientist Berzelius in 1836 as a chemical substance added in small quantities to the reacting medium to increase the rate of a chemical reaction by reducing the energy needed to reach the activated complex phase in which the reactants move through their molecular orbitals and then decompose to produce the products. The catalyst does not cause thermodynamically impossible reactions but rather accelerates possible reactions. It has been found that adding small amounts of metallic materials, such as transition elements, to the centre of some reactions increases reaction rates compared to the absence of these materials [1]. Pyran is a heterocyclic, unsaturated (non-aromatic) hexacyclic compound consisting of six main atoms, five of which are carbon atoms and one oxygen atom [2], and has the chemical formula C5H6O, Its systematic names (IUPAC) are 2H-Pyran, and 4H-Pyran, and there are other names: H-Oxine, 4H-Oxine2. There are isomers of pyran, with different positions of the double bond. In 2H-pyran, the saturated carbon is at the 2-position, while in 4H-pyran, the saturated carbon is at the 4-position [3].



They have a variety of fascinating biological functions and significant significance in organic biochemistry [4]. The presence of 4H-cyanohydrins and their derivatives in a large variety of physiologically active chemicals makes them a significant class of molecules. Applications of 4H-cyanopyran in modern pharmaceutical chemistry include antioxidant and antibacterial properties [5]. anticoagulant, antiallergic [8], antituberculosis [6], and antitumor [7].

2. Materials and Methods

2.1 Chemical Used, Chemicals prepared by Aldrich, BDH Thomas, Fluka, and Merck were used

2.2 Instruments Used, The melting point is measured using a thermometer 9300, KBr disk with a scale of 400-4000 cm-1, FT-IR 8400S Shimadzu spectrophotometer, 1H- and 13C-NMR spectra of Bruker equipment operating at 400 MHz. Thin-layer chromatography (TLC) was analysed using 0.2 mm thick Fluka silica gel plates**.**

2.3 Preparation of catalyst derivatives. [9]
Weigh (1 mol) potassium nitrate and (3 mol) alumina and grind them evenly in a mortar, then add 5 drops of deionized water during the grinding process until uniform, form a paste, and dry at 100 °C. It is activated at a temperature of 110 degrees Celsius for an hour, then the mixture is placed in a ceramic bowl and activated at a temperature of 600 degrees Celsius for 3 hours. The catalyst was left to cool in the air.

2.3 Preparation of 2-amino pyran derivatives (M36-M40).[10,11]
Dissolve (0.015 mol) chalcone in (15 ml) ethanol in a circular flask (15 ml) equipped with a magnetic stirrer, then add (0.225 mol, 0.148 ml) malononitrile and stir for (15) minutes (11), and add the catalyst (Al2O3-OK). 20% wt chalcone) Stir in a water bath at (40)°C for 3-4 hours. The solution was then filtered, the precipitate was discarded, and the filtrate was allowed to dry and recrystallized from absolute ethanol. Use TLC plates to track the reaction process. Table (2-10) shows in Table (MH36-MH40).

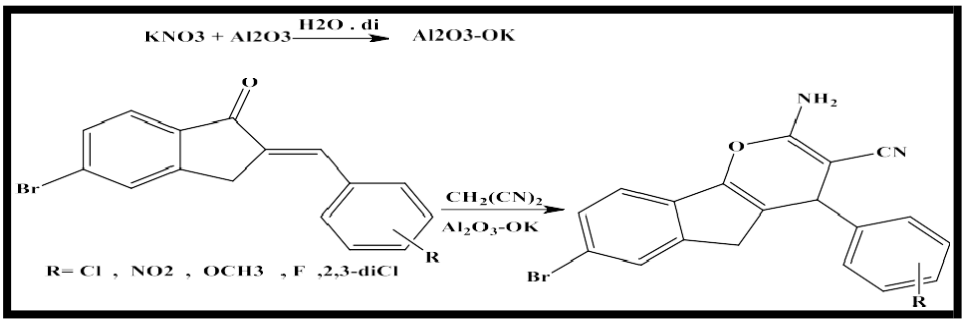
Comp. No.	R	**Molecular formula**	**m.p. °C**	**Yield %**	**Colour**
MH_{16}	**4-Cl**	**$C_{19}H_{12}BrClN_3$**	**165-167**	**42**	**Off white**
MH_{17}	**4-NO_2**	**$C_{19}H_{12}BrN_4O_2$**	**183-185**	**38**	**Brown**
MH_{18}	**4-OCH_3**	**$C_{20}H_{15}BrN_3O$**	**116-118**	**40**	**Blue**
MH_{19}	**4-F**	**$C_{19}H_{12}BrFN_3$**	**207-209**	**46**	**Light green**
MH_{20}	**2,3-diCl**	**$C_{19}H_{11}BrCl_2N_3$**	**143-145**	**57**	**Orange**

2.4 Biological activity study

Staphylococcus aureus is both gram-negative and gram-positive. The pathogen used in this study is Escherichia coli. The Department of Pure Science and Life Sciences Education uses Molton-Hinton agar as a bacterial growth medium. Chemical solutions of M36, M37, M39, and M40 were prepared using dimethyl sulfoxide (DMSO) at concentrations of (0.01, 0.001, 0.0001) mg/mL [12, 13]. Determine and monitor the minimum inhibitory concentration (MIC). Mueller Hinton agar was used as a nutrient medium and diffusion techniques were performed to confirm the susceptibility of the bacterial isolates used in the study. After preparing the culture medium, sterilize it, distribute it onto Petri dishes, and allow it to freeze. Next, drill four small holes in each plate. They were then incubated at 37°C for a full day. Derivatives used. These indicate the sensitivity of the derivatives used. As the diameter increases, these derivatives depend on the damping diameter of the plate surrounding the hole used. When the chemical produced shows an inhibitory effect, its biological activity increases, similar to the inhibitory effects of antibiotics. [14,15].

3. Results and Discussion

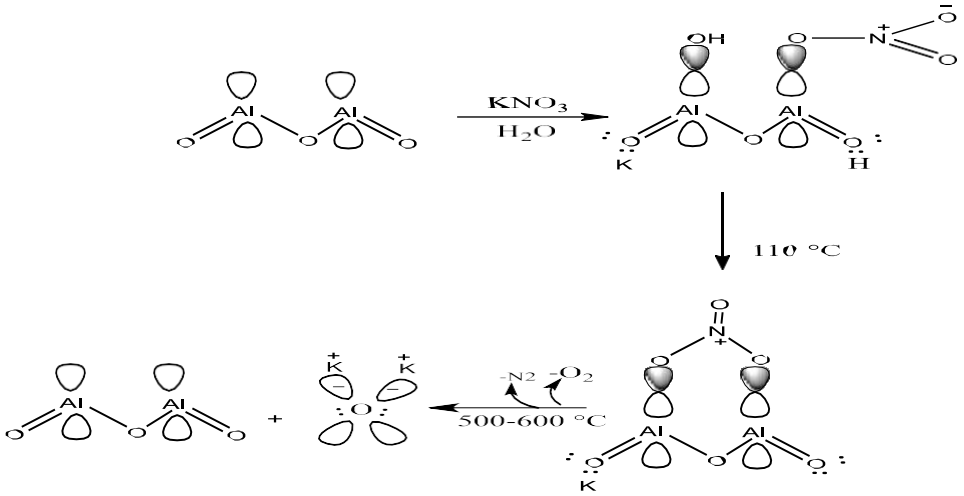
2-Aminopyran compounds were prepared according to the following scheme



Scheme (1): Path of the Ready Compounds (M36-M40)

3.1 Characterization of catalyst

The proposed mechanism for preparing the catalyst was as follows:



Scheme; 2 Mechanism of catalytic reaction

SEM analysis of the catalyst shown in Figure 1 shows that the cross-sectional area (2 μm) and peak radius of the catalyst particles is 2.529 nm belonging to nanocrystals [16].

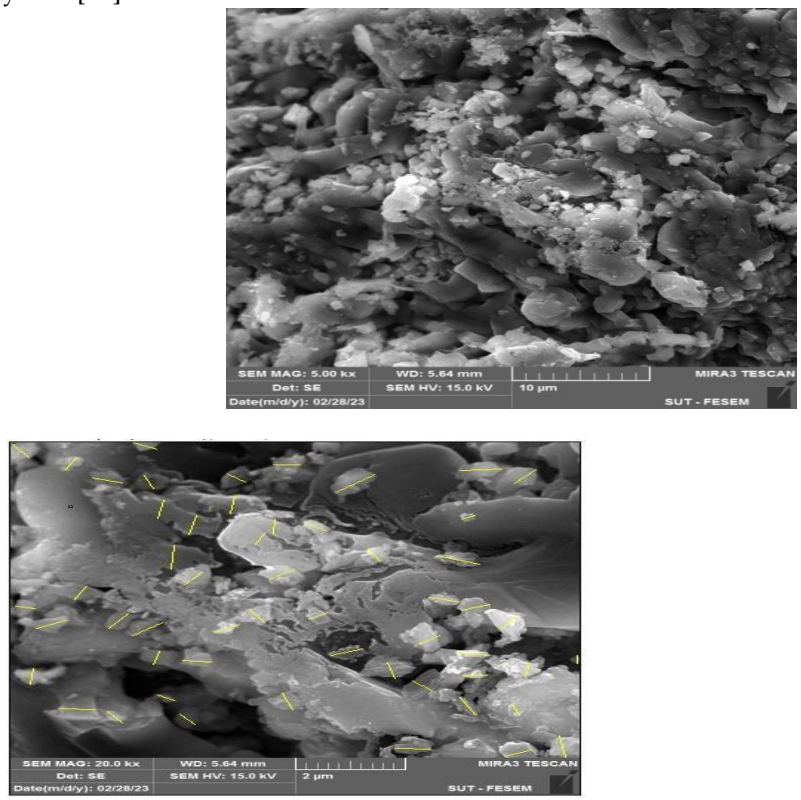
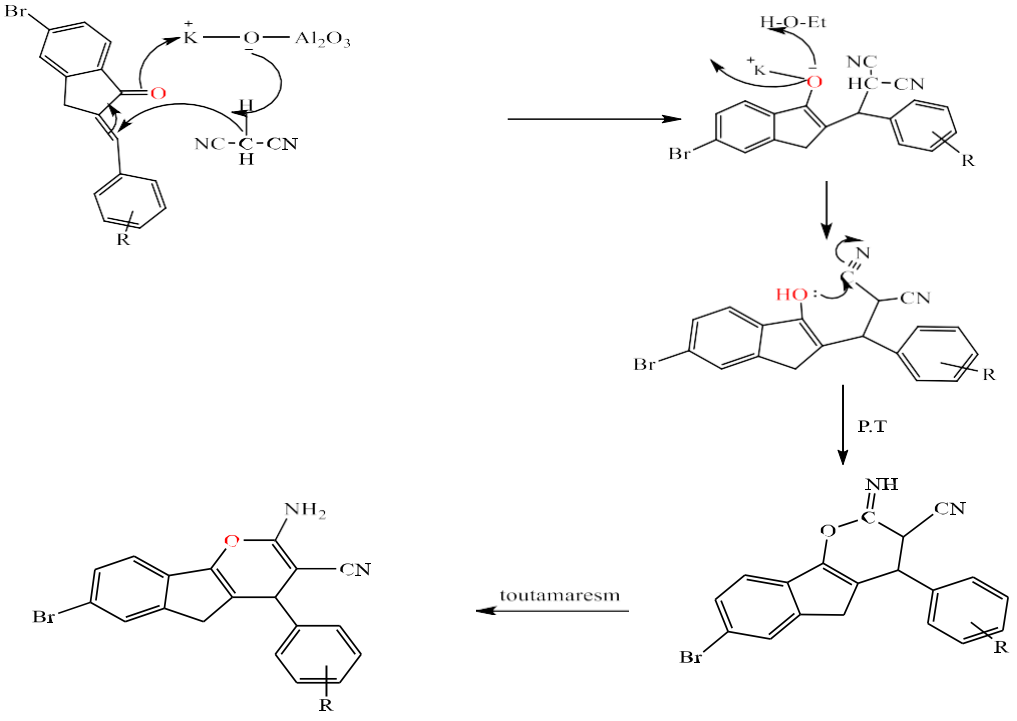


Figure (1): shows an SEM image of the catalyst

3.2 Characterization of 2- Amino pyran derivatives (M36-M40)

The proposed mechanism for preparing the 2-AMINO PYRAN was as follows:



R= 4-Cl ,4-NO2 , 4-OCH3 , 4-F , 2,3-diCl

Scheme (3): Mechanism of preparation of 2-aminopyran derivatives [MH36- MH40]

Examining the 2-aminopyran derivative's infrared spectrum (FT-IR) revealed two absorption bands that belonged to the bond (NH2) and appeared in the interior in the ranges of (3240-3311) cm-1 and (3320-3375) cm-1. Additionally, an absorption band in the range of (3047-3078) cm-1 also appeared as a result of the aromatic bond's expansion. Additionally, it was noted that absorption bands appeared of (2168-2216) cm-1 due to the expansion of aromatic bonds (CN) and (1583-1595) cm-1 due to the expansion of the alkene affinity band (C=C); an absorption band appears in the range of (1360-1390) cm-1 due to the stretching of the affinity group (CO-O); and two absorption bands appear in the ranges of (2937-2962) cm-1 and (2824-2910) cm-1 that is associated with the stretching of the aliphatic bond (C-H) as demonstrated [16]. Table (3-9).

Table (2): Infrared absorption results (cm-1) for [MH36-MH40] compounds

		IR (KBr) cm^{-1}						
Comp. No.	**R**	**ν(C-H) Arm**	**ν(C-O)**	**ν(C-H) .Aliph.**	**ν(NH_2)**	**ν(C=C)**	**ν(C=C) Arom.**	**Others**
MH_{36}	**4-Cl**	3060	1390	2945 2844	3371 3311	1589	1512 1463	ν(C-Br) 622 ν(CN)2179
MH_{37}	**4-NO_2**	3078	1377	2937 2824	3345 3240	1588	1519 1454	ν(C-Br) 654 ν(CN)2168
MH_{38}	**4-OCH_3**	3047	1360	2939 2841	3325 3260	1595	1515 1490	ν(CBr)609 ν(-CN)2196
MH_{39}	**4-F**	3051	1384	2950 2896	3375 3303	1583	1514 1463	ν(C-Br) 667 ν(CN)2216
MH_{40}	**2,3di-Cl**	3054	1385	3962 3910	3320 3244	1593	1537 1487	ν(C-Br) 619 ν(CN)2184

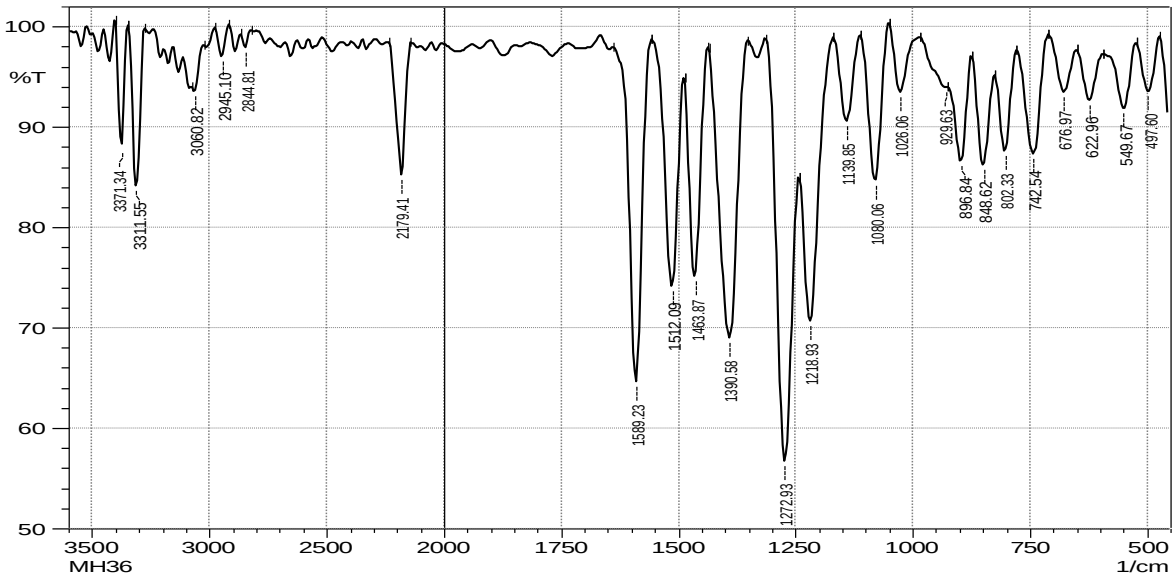


Figure (2): The infrared spectrum of the compound [MH36]

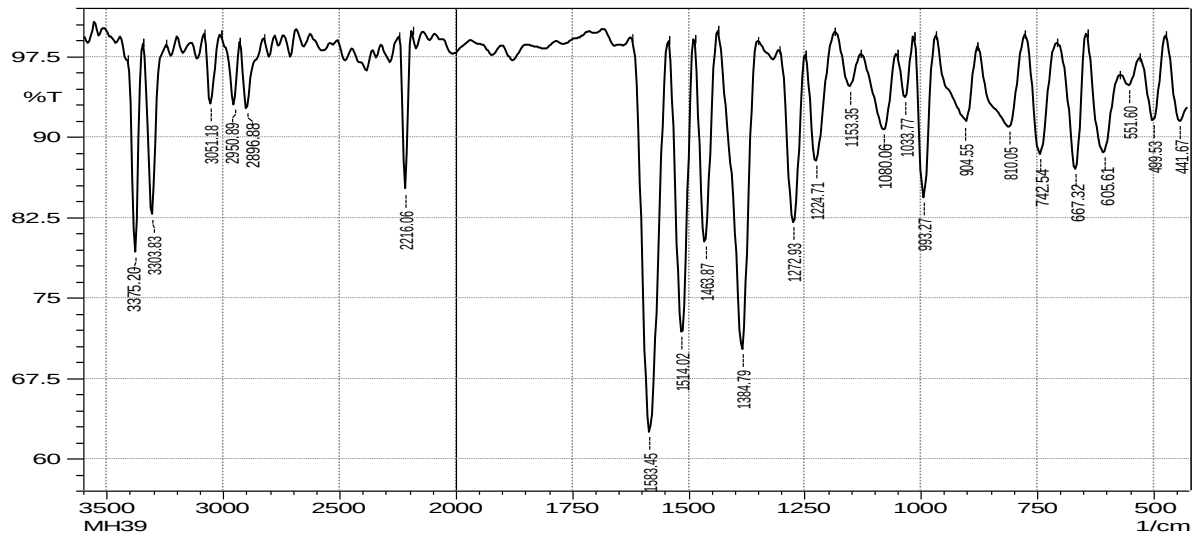


Figure (3): The infrared spectrum of the compound [MH39]

The 1H-NMR spectrum of the compound [MH36] shows that multiple signals attributed to the aromatic ring protons appear in the ppm range (7.18-7.91), and a signal at the ppm position (4.06) attributable to the aromatic ring protons that is consistent with the aromatic group proton appears The pyran ring (CH) attached to the ring has a signal at ppm (6.84) attributed to the protonation of the (NH2) group, and the signal appears at. The signal appears at the ppm site (3.40) attributed to the proton of the group (2CH) attached to the aromatic ring (3.40) and at the ppm site (2.51) attributed to the solvent (DMSO-d6), as shown in Figure 4

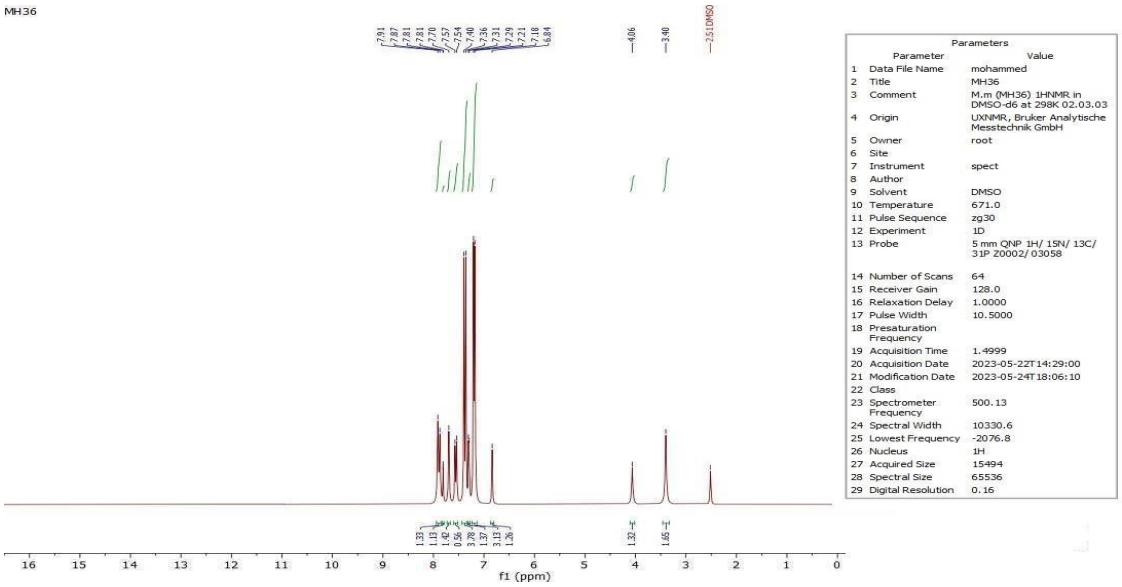


Figure (4): The HNMR spectrum of the compound [MH36]

The signal for compound [MH36] may be seen in its 13 C-NMR spectrum at a ppm point (147.35), which is located distant from the group carbon (=CO-O) of the compound. The signal that occurs at the ppm location (159.61) is assigned to the group carbon (=C-NH2), the signal that appears at the ppm position (122.76-157.86) is credited to the two ring

carbons, and the group carbon (CN) is responsible for the value of 120.28.and the signal at ppm position (112.58) is attributed to the carbonyl group (=C) of the double bond in the pyran ring, except at ppm position (28.84), where there is an additional signal attributable to the carbon group (CH2) and the carbon of the pyran ring attached to the benzene ring at ppm position (49.02). The signal appearing in the ppm range (39.48 - 40.46) is attributed to the carbonation of the solvent (DMSO-d6).

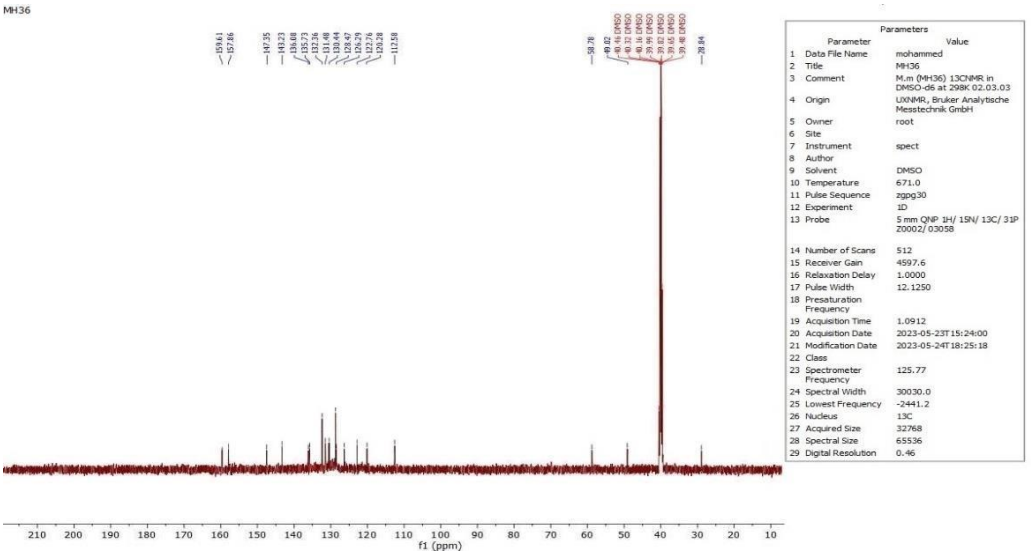


Figure (5): The 13C-NMR spectrum of the compound [MH36]

3.3 Evaluation of the Biological Activity of Prepared Compounds

These bacteria were chosen for their medical importance, as they cause many diseases and have different resistance to antibiotics. The bioavailability of many of the prepared compounds was evaluated using etching methods and antibiotic-level measurements [17,18]. The results showed that different ratios of Gram-positive and Gram-negative compounds could inhibit the growth of bacteria, as shown in Table 3 [19,20].

Comp. No.	E. Coil Conc. mg/ml			Staph. Aureus Conc. mg/ml		
	0.01	0.001	0.0001	0.01	0.001	0.0001
M36	15	10	5	18	10	0
M37	14	11	7	13	8	0
M39	18	16	11	10	5	5
M40	13	13	5	15	10	5
Amoxicillin	22	17	16	20	19	15

4. Conclusion

The use of catalysts in reactions is safer than the use of other chemicals and is more available and easy to obtain. The compounds also showed high purity in their diagnosis. In addition, they showed good effectiveness against two types of Gram-negative and Gram-positive bacteria\

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