

Synthesis and Characterization of Mannich Base Derivative from Benzimidazole

Muhammed Khidir Aljobory, Nawfal Sheet Mohammad, Hussein Yousif Ridha

Ministry of environment, Department of protection and improvement of environment, northern region, Kirkuk, Iraq.

Department of Chemistry, College of Education for Pure Science, University of Mosul, Mosul, Iraq.

Department of Chemistry, College of Education for Pure Science, University of Mosul, Mosul, Iraq.

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Abstract

The research involved the preparation of several new organic compounds (Mannich bases) derived from the reaction of equimolar amounts of p-chlorobenzaldehyde with benzimidazole and a number of the following primary amines (aniline, 4-hydroxyaniline, 4-chloroaniline, 4-methoxyaniline, 4-methylaniline, 4-aminopyridine, 3-acetylaniline, 2-aminothiazole, N-(4-amino phenyl)acetamide and 2-methyl-4-amino pyridine). Ethanol was used as the solvent, and the synthesized compounds were proved by infrared (IR) spectroscopy and (NMR) spectroscopy for proton and carbon. Elemental analysis was also performed using an elemental analyzer (EA Euro vector 3000), and the finishing point of the reaction was monitored and verified by thin-layer chromatography (TLC).

Keywords: *Benzimidazole; Mannich base; primary amine.*

Introduction

The Mannich bases have been classified as famous compounds long time ago and still as a chemical tool in these compounds continues. Because of its efficiency in pharmacological aspects and their industrial requests such as polymers used in paint [1,2]. The term of the Mannich base includes all organic compounds containing the active group (CH_2N). Carl Mannich was the first to study Mannich bases; consequently, the name of these compounds is associated with him. Since then, Mannich bases have become a significant subject of study and research. Scientists such as Hellmann, Opitz and Reichert [2] focused on Mannich bases and their applications across various fields, ultimately developing diverse chemical methods for their synthesis. Mannich reaction is a nucleophilic addition reaction to the carbonyl group. The kidney alkylation of the amines by a compound containing an effective proton and a carbonyl group with primary or secondary amine or ammonia sometimes [3]. The result is β -amino carbonyl or one of its derivative methyl amino the Mannich bases are called [4,5]. In the industrial field and because of high selectivity and sensitivity complexes of the Mannich bases for metal ions where they proved their ability to remove toxic solids especially those polluting the environment [6]. Mannich is also can be used in the synthesis of medicinal compounds e.g. rrolitetracycline, fluoxetine, tramadol and tolmetin [7-8], hypertensive [9], anticancer [10], and antibacterial activities [11]. Some of these compounds contain an amide group, which possesses a significant ability to form complexes with metal ions. This occurs through heteroatoms such as oxygen, nitrogen, and others that can bind to metal ions to form complexes with Mannich bases. Other industrial applications include polymers used in coatings and paints. They can also be used in the treatment of industrial waste, particularly in the removal of toxic heavy metals, which have harmful effects on animal and plant life. These toxic heavy metals include mercury, lead, copper, cadmium, and zinc [12]. In 2004, researchers succeeded in synthesizing certain Mannich bases through the condensation of morpholine, semicarbazide, and benzaldehyde; the resulting compounds demonstrated the ability to form complexes with various metal elements. In 2005, researchers prepared numerous Mannich bases from compounds containing active hydrogen atoms by reacting them with aromatic secondary amines and aromatic

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aldehydes. These synthesized compounds were deemed significant due to their biological applications and broad industrial importance, and they were utilized as intermediates in polymer chemistry. The aim of this research is to prepare the number of Mannich bases from the interaction of equal moles of both 4-chlorobenzaldehyde with benzimidazole and primary amine.

Materials and Methods

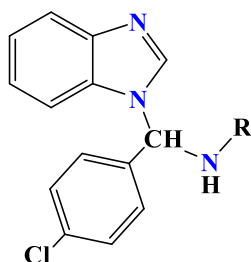
The Melting points It has been determined by open tube capillary method and are uncorrected electro thermal Melting Point Stuart (SMP 10), IR spectra were obtained on a sidco biotech FT-IR 600 spectrometer. ^1H -NMR and ^{13}C -NMR spectra (Bruker advance 400 MHz spectrometer). Elemental analysis was performed on some compounds, and the proportions of the elements within them were found to be very close to the calculated values. progress of The reactivity and purity of the products were verified by thin layer chromatography (TLC).

Experimental part

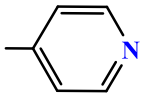
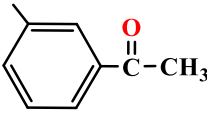
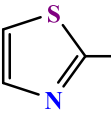
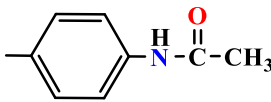
General preparation method :

The solution of Benzimidazole (0.01 mole) in the least amount of ethanol was mixed with 4-chlorobenzaldehyde (0.01 mole) put the mixture in an ice bath with continuous stirring for one hour, Then add a cold lotion solution from one of the primary amines (0.01 mole) Dissolved in ethanol and refluxed for (6 hr), completion of the reaction It has been monitored by (TLC), The evaporation of the solvent is then a precipitation, washing the residual using cold ethanol, crystallization from Suitable solvent. The properties of compounds show in the table (1).

Table (1) : Physical data of preparation compounds



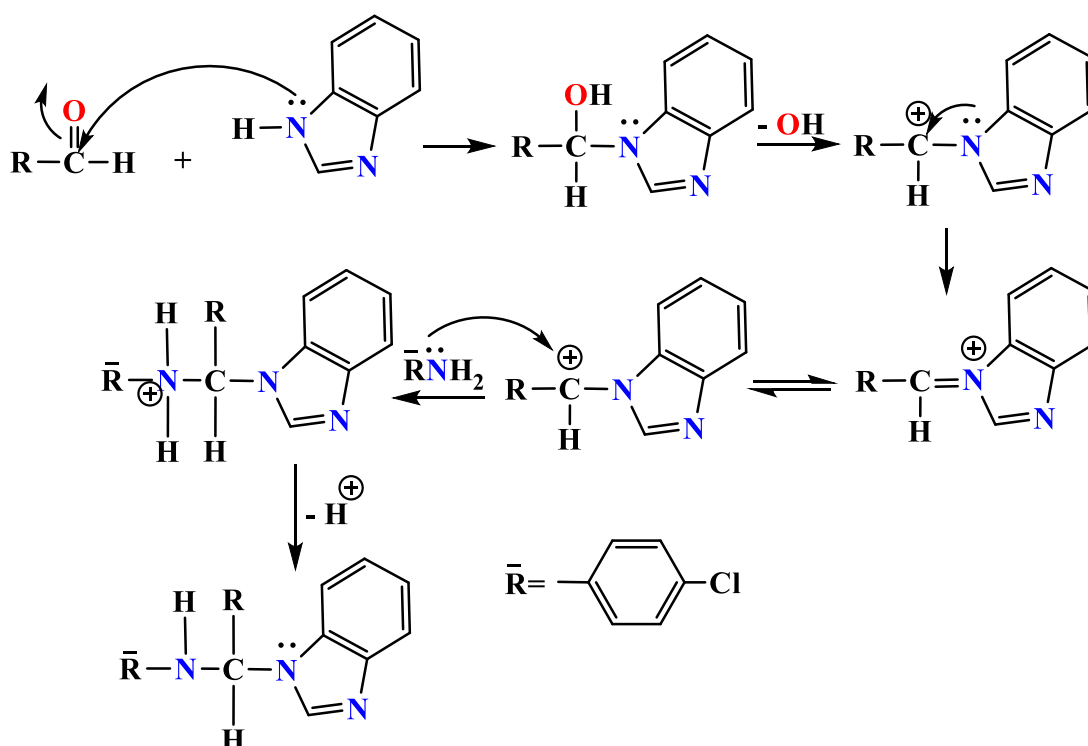
Comp. No.	R	Molecular formula	m.p °C	Yield%	Color	Recrst. solvent
1		$\text{C}_{20}\text{H}_{16}\text{ClN}_3$	132-134	65	Pink	Ethanol
2		$\text{C}_{20}\text{H}_{16}\text{ClN}_3\text{O}$	145-146	47	Yellow	Ethanol
3		$\text{C}_{20}\text{H}_{15}\text{Cl}_2\text{N}_3$	222-224	61	Brown	Ethanol
4		$\text{C}_{21}\text{H}_{18}\text{ClN}_3\text{O}$	196-198	58	Yellow	Ethanol
5		$\text{C}_{21}\text{H}_{18}\text{ClN}_3$	248-250	74	Dark Brown	Ethanol

6		$C_{19}H_{15}ClN_4$	180-182	86	Orange	Dioxane
7		$C_{22}H_{18}ClN_3O$	261-263	38	white	Ethanol
8		$C_{17}H_{13}ClN_4S$	150-152	70	Brown	Ethanol
9		$C_{22}H_{19}ClN_4O$	235-237	52	white	Dioxane
10		$C_{20}H_{17}ClN_4$	118-120	62	Yellow	Dioxane

Results and discussion

Vehicles were prepared (1-10) Benzimidazole condensation with 4-chlorobenzaldehyde and primary amine using absolute ethanol as the medium of the reaction as shown in the following mechanical :

The proposed reaction mechanism start with a nucleophilic attack of the pair electron of the secondary amine atom to electrophilic carbonyl carbon of the aldehyde, accompanied by displacement of the carbonyl π -electrons toward the oxygen atom to form a carbinolamine intermediate. Subsequently proton transfer and dehydration lead to the creation of an iminium ion. The iminium species suffers from nucleophilic attack by the amine (RNH_2), followed by deprotonation to afford the final neutral product. Overall, the reaction proceeds through nucleophilic addition, proton transfer, dehydration, iminium formation, nucleophilic attack, and deprotonation



Reaction mechanism

Interaction was confirmed following the change in physical properties of melting point and color as well as IR charts and NMR for proton and carbon, as the spectra showed (N-H) within range ($3107\text{--}3266\text{ cm}^{-1}$), also the peak at ($2968\text{--}2986\text{ cm}^{-1}$) for (C-H) aliphatic, peak at ($3022\text{--}3068\text{ cm}^{-1}$) for the (C-H) aromatic, In addition to that the stretching banding at ($1487\text{--}1527\text{ cm}^{-1}$) is assigned for C=C. In addition to the appearance of frequency at ($1224\text{--}1238\text{ cm}^{-1}$) for (C-N)[13,14], showed results that confirm our expectation as mention in table (2).

Table (2) : IR of synthesized compounds

Comp. No.	FTIR vcm^{-1} (KBr)						
	N-H	C-H Aliph	C-H aroma	C-N	C=C	C=N	Others
1	3168	2976	3028	1236	1487	1583	808 (C-Cl)
2	3245	2968	3032	1224	1504	1608	3410 (O-H)
3	3214	2986	3046	1236	1496	1604	732 (C-Cl)
4	3188	2980	3058	1228	1510	1598	as.1280(C-O-C) sy.1140(C-O-C)
5	3156	2972	3060	1232	1492	1588	-
6	3266	2984	3022	1238	1488	1614	-
7	3260	2979	3060	1232	1520	1610	1672 (C=O)
8	3107	2978	3043	1234	1527	1616	746 (C-Cl) 615 (C-S-C)
9	3204	2970	3068	1230	1526	1584	1645 (C=O)
10	3176	2974	3026	1238	1498	1590	-

$^1\text{H-NMR}$ spectrum for compound (9), The following explains characteristic chemical shifts, (DMSO- d_6) ppm. (2.17 ppm) for (CH_3), (6.02 ppm) for (CH), (7.41 ppm) (NH-CH), (9.26 ppm) (NH-C=O), (6.28-7.05 ppm) for (phenyl-Cl).

$^{13}\text{C-NMR}$ spectrum for compound (10), The following explains characteristic chemical shifts, (DMSO- d_6) ppm. (23.56 ppm) for (CH_3), (99.10 ppm) (CH), (161.57 ppm) (N=C-CH_3), (134.46 ppm) (C-Cl), (153.94 ppm) (C-NH), (139.95 ppm) (C=N Benzimidazole).

Table (3) : C.H.N. analysis of prepared compounds of (1,4,8,9)

Comp. No.	Molecular weight	C%	H%	N%	O%
1	333.82	Calc.71.96	4.83	12.59	-
		Found.71.62	4.78	12.34	-
4	363.85	Calc.69.32	4.99	11.55	4.40
		Found.69.12	4.86	11.39	4.52
8	340.83	Calc.59.91	3.84	16.44	-
		Found.59.77	3.90	16.30	-
9	390.87	Calc.67.60	4.90	14.33	4.09
		Found.67.68	4.82	14.40	4.22

Conclusion

In this study, a novel Mannich base derivative of benzimidazole was successfully synthesized through a Mannich condensation reaction. The synthetic procedure provided the desired derivative in a satisfactory yield, demonstrating the suitability of the selected reaction conditions for the preparation of benzimidazole-based Mannich compounds.

The synthesized compound was proved using appropriate spectroscopic and analytical techniques, Inclusive FT-IR, ^1H -NMR, and ^{13}C -NMR spectroscopy. The obtained spectral data were consistent with the proposed molecular structure and confirmed the formation of the expected functional groups and the successful incorporation of the aminomethyl moiety into the benzimidazole framework.

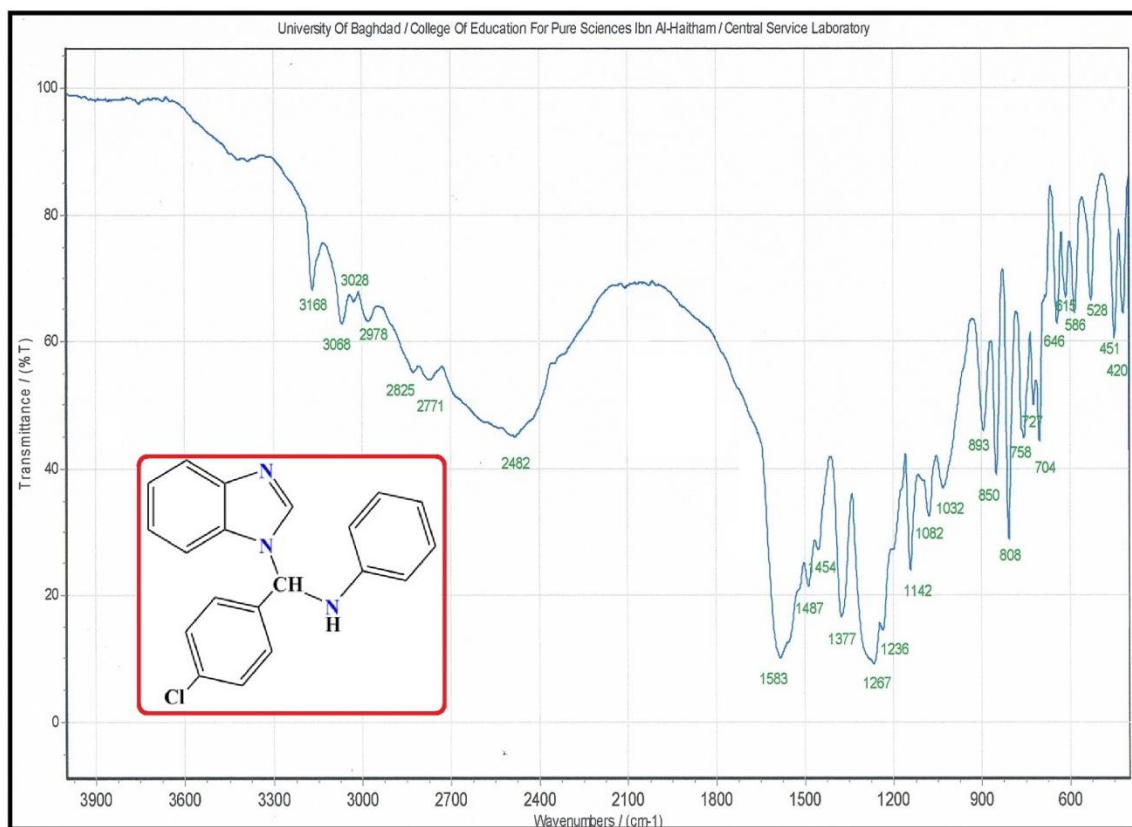


Fig.1. IR spectrum of compound (1)

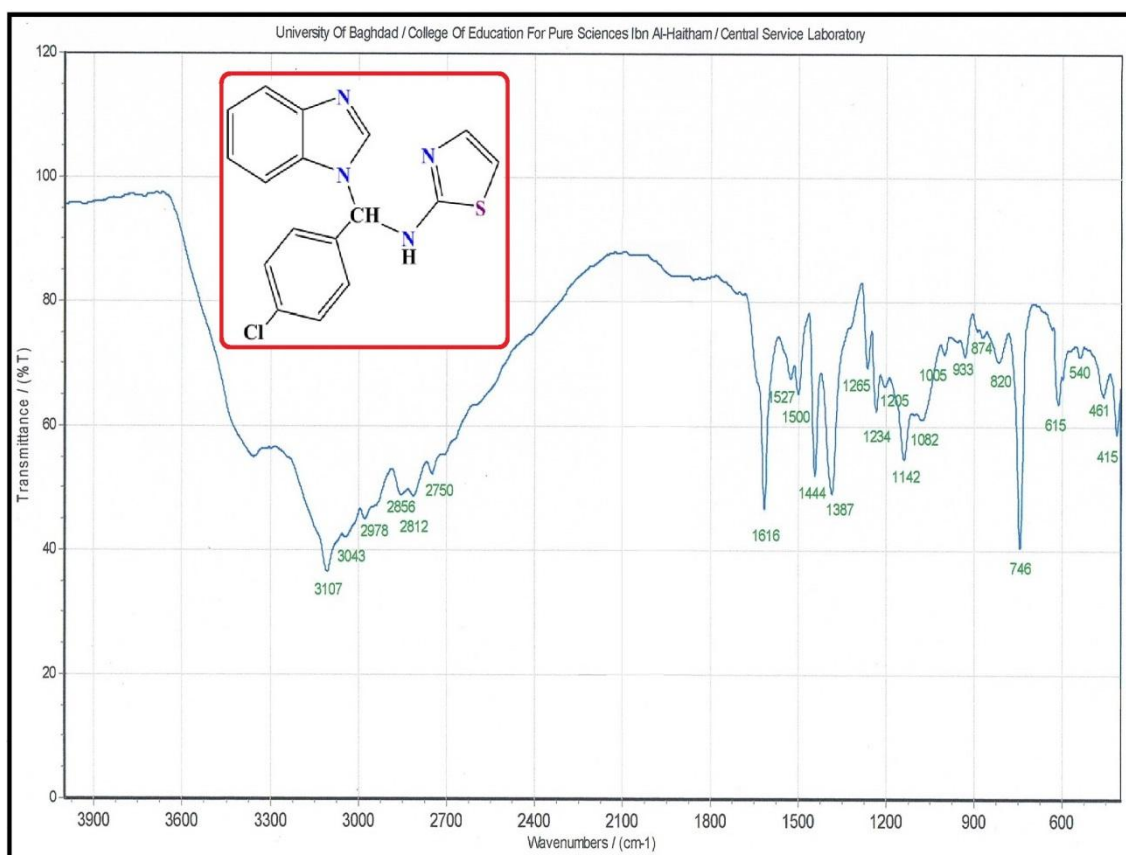
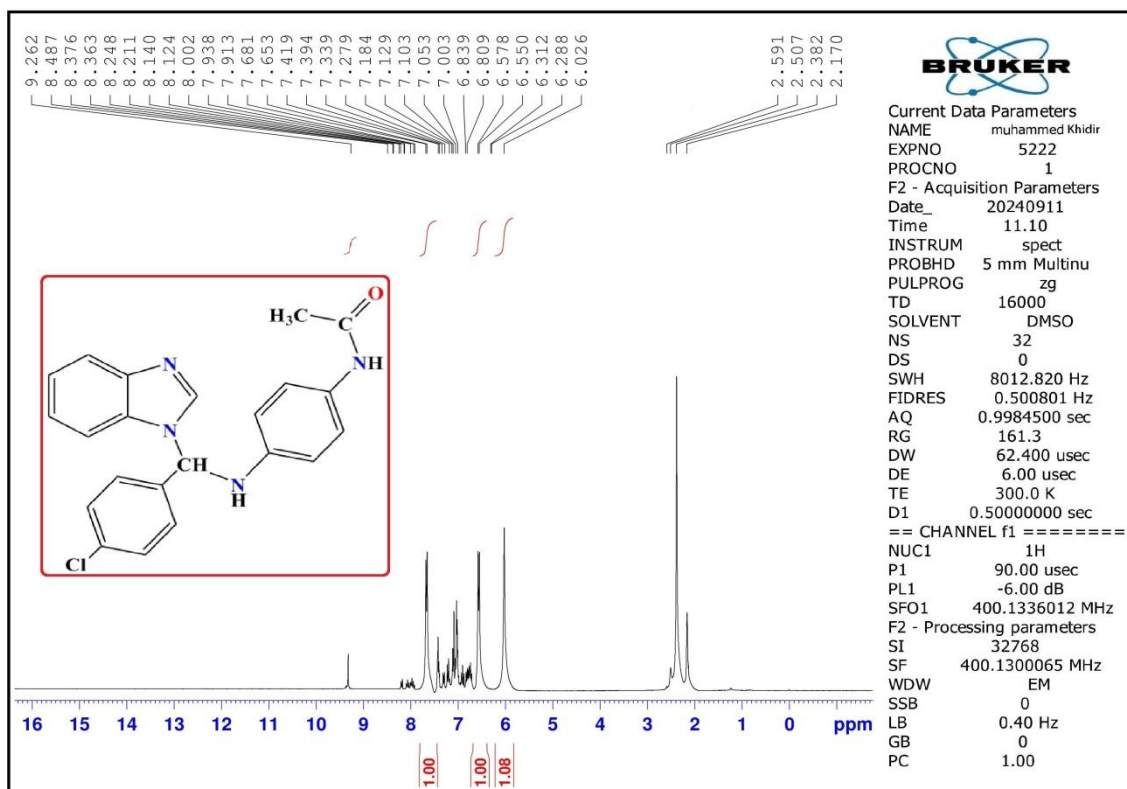
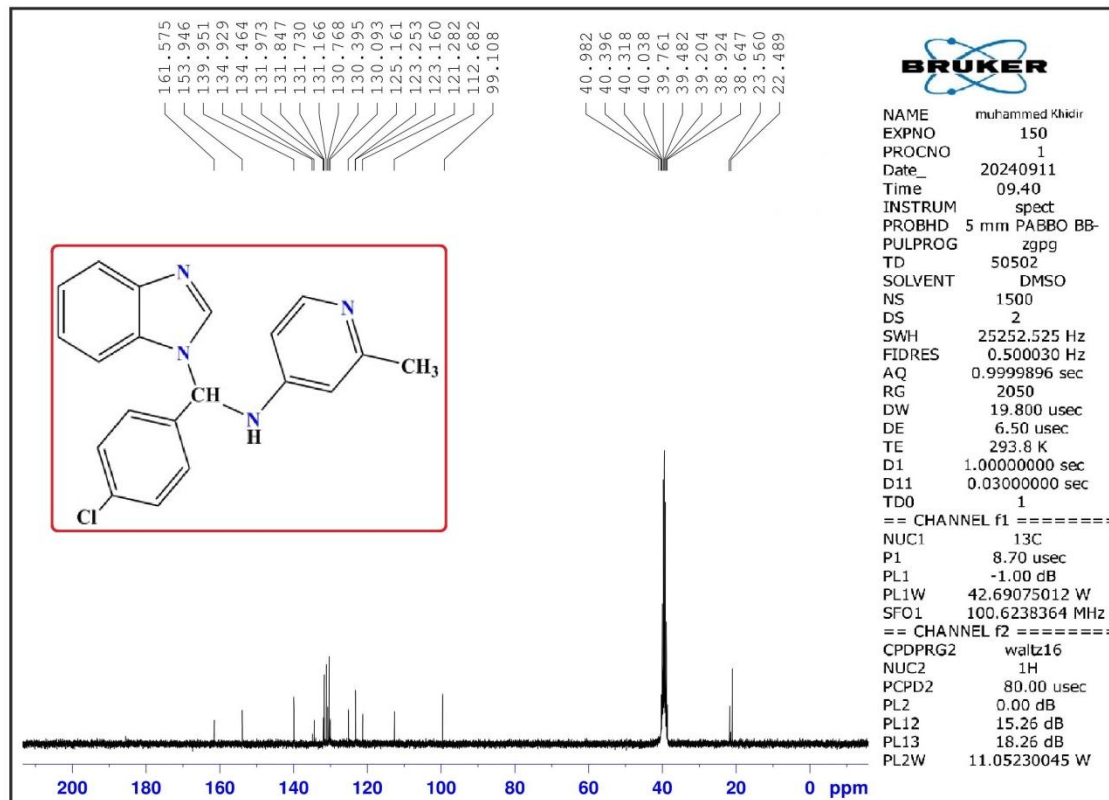


Fig.2. IR spectrum of compound (8)

Fig.3. ¹H-NMR spectrum of compound (9)Fig.4. ¹³C-NMR spectrum of compound (10)

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