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Design, synthesis and antimicrobial activity screening of new dimethyl maleimidyl heterocyclic derivatives based on 4-(N-dimethyl maleimidyl) phenyl hydrazide

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Abstract: The research involved the design and synthesis of a collection of innovative dimethyl maleimide heterocyclic derivatives through a multistep synthesis process. The synthesis of compound [1] ethyl-4-(N-dimethyl maleimidyl) benzoate was achieved through the reaction of dimethyl maleic anhydride with ethyl-4-amino benzoate. In the second stage, molecule [1] was reacted with hydrazine hydrate to provide compound [2] 4-(N-dimethyl maleimidyl)phenyl hydrazide. Compound [2] is the crucial precursor from which all objectives compounds were produced. In the third step, compound [2] was subjected to a condensation reaction with aromatic aldehydes, yielding Schiff base derivatives [3-5] which were subsequently reacted with acid to produce target compounds [6-8] specifically dimethyl maleimides featuring dihydroquinazoline cycles. Meanwhile, the treatment of compounds [3-5] with acetic anhydride resulted in additional target compounds [9-11] namely dimethyl maleimides containing oxadiazole cycles. Furthermore , the introduction of compound [2] in a reaction with CS2 in a medium that is basic, followed by treatment with hydrazine hydrate, resulted in the formation of the target compound [12] while the condensation of compound [2] with pyrrole carbaldehyde produced the target compound [13]. The results of the antimicrobial efficacy research of the target compounds against four species of bacteria and fungi are quite encouraging.

Keywords: *Dihydroquinazoline, Dimethyl Maleimide, Oxadiazole, Schiff Base, Triazole.*

1. Introduction

Cyclic imides and their derivatives represent a valuable group of bioactive compounds with wide range of various important applications [1, 2]. Besides cyclic imide moiety is an important core present in many drugs and medicines because to their extensive array of biological activities inclualing antimicrobial [3] antifungal analgesic [4] antioxidants [5] antinflammatory [6] and anticancer activity [7].

On the other hand heterocyclic compounds are very important moieties in organic chemistry and since they exhibit a broad range of biological actions, including anticancer and antiviral properties, analgesic and anti-inflammatory they are used as pharmaceuticals, in drug synthesis, veterinary products , agrochemicals , antioxidants and as corrosion inhibitors [8, 9].

Depending all these points we thought that it was too worthy on to design new molecules that contain these two active components (imide rings and hetero cycles together). Consequently, the current study entailed the synthesis of novel dimethyl maleimide derivatives containing different heterocycles based on 4-(N-dimrthyl maleimidul)phenyl hyrazide [14], through applying of different synthetic methods with high expectations that these new compounds will exhibit high biological activity and give promising results.

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2. Experimental

The melting temperatures of the synthesized compounds were determined using a Gallen Kamp melting point equipment and were reported uncorrected. FTIR spectra were acquired using a Shimadzu FTIR-8400 Fourier Transform spectrophotometer. The Ultrashield 400MHz Bruker apparatus was employed to record both 1H-NMR spectra in the presence of the solvent DMSO-d6 and the internal standard tetramethyl silane.

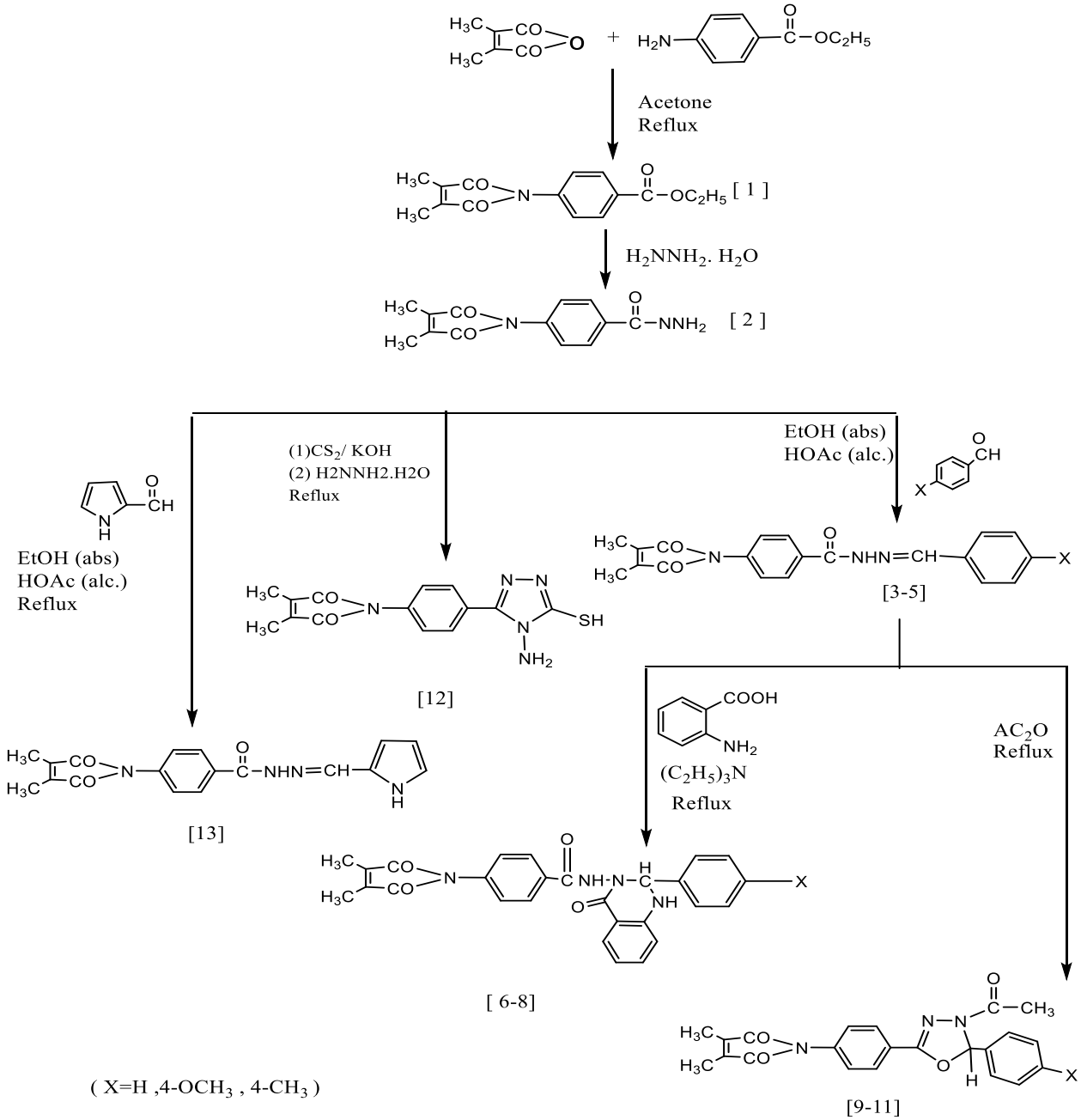

Figure 1.
Synthetic steps of compounds [1-12].

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2.1. Synthesis of Ethyl-4-(N-Dimethyl Maleimidyl) Benzoate [1]

To the solution of dimethyl maleic anhydride (0.01mol, 1.26g) dissolved in (15mL) acetone a solution of (0.01mol, 1.65g) ethyl-4-aminobenzonte dissolved in (15 mL) acetone was added drop by drop with good stirring and cooling [10]. After completion of the addition, the amalgamation was agitated at ambient temperature for two hours. Subsequently, the resultant precipitate was then put to filtration,dried, and purified through recrystallization using ethanol.

2.2. Synthesis of 4-(N-dimethyl maleimidyl)phenyl hydrazide [2]

The chemical [2] was synthesized via the reaction of compound [1] (0.01 mol, 2.73 g) with hydrazine hydrate (0.015 mol, 0.75 g) over a period of four hours. Subsequently, 20 mL of ethanol was introduced, and reflux conditions were sustained for an additional eight hours [11]. Following the reflux process, the mixture was allowed to cool. The resultant precipitate was thereafter filtered and rinsed with distilled water, dried, and subsequently underwent recrystallization using dioxane.

2.3. Synthesis of 4-(N-dimethyl maleimidyl)-N-benzylidene benzamide [3-5]

The designated Schiff bases [3-5] were produced by refluxing a mixture of compound [2] (0.01 mol, 2.59 g) and 0.01 mol of aromatic aldehyde (benzaldehyde, p-anisaldehyde, p-tolualdehyde) in 25 mL of pure ethanol, with a few drops of glacial acetic acid as a catalyst [12, 15] for eight hours. Upon completion of the reflux, This mixture was cooled, and the resultant material was filtered, dried, and subsequently recrystallized using a suitable solvent [16, 17].

2.4. Synthesis of 2-(Substituted Phenyl)-3-[N-(4-(N-dimethyl Maleimidyl) Benzamido]-Dihydroquinazoline-4-One [6-8]

The titled compounds [6-8] were synthesized through reaction of anthranilic acid (0.01mol,1.37g) with (0.01mol) of schiff bases [3-5] in dioxane with few drops of triethylamine with stirring and cooling in ice bath (20 min) [11, 18]. The resulting mixture underwent reflux for ten hours, followed by cooling, subsequently, the precipitate underwent filtration, drying, and recrystallization using a suitable solvent.

2.5. Synthesis of 5-(4-(N-Dimethyl Maleimidyl)Phenyl-3-Acetyl-2- (Substituted Phenyl)-1,3,4-Oxadiazole [9-11]

The titled compounds [9-11] were synthesized via refluxing the mixture of schiff bases [3-5] (0.01 mole) and (15mL) of acetic anhydride for six hours .The resultant mixture after reflux was permitted to cool to the ambient temperature before being introduced into chilled water while stirring, which continued for thirty minutes [9]. The precipitate underwent filtration, was washed once more with water, dried, and subsequently recrystallized using a suitable solvent.

2.6. Synthesis of N-[4-(4-Amino-5-Mercapto-1,2,4-Triazole-3-y1) Phenyl Dimethyl Maleimide [12]

The designated compound [2] (0.01 mol, 2.59 g) was dissolved in 20 mL of absolute ethanol containing(0.01 mol ,0.56 g) of KOH, to which (0.02 mol ,1.52 g) of quantity of carbon disulfide was introduced while stirring vigorously. The mixture underwent reflux for one hour, following which the resultant solid was filtered. Following drying, it was refluxed with (5 mL) of distilled water and(0.01 mol ,0.48 mL) of hydrazine hydrate for eight hours [11, 19]. Upon completion of the reflux, the resulting mixture was permitted to cool, subsequently neutralized with HCl, The resulting powder was subjected to filters, followed by washing with distilled water, drying, and recrystallization from ethanol.

2.7. Synthesis of 4-(N-Dimethyl Maleimcidyl)-N-(2-Imino Pyrrol) Benzamide [9].

The melding of compound [2] (0.01 mol, 2.59 g) and Pyrrol-2-aldehyde (0.01 mol, 0.95 g) in (20 mL) of absolute ethanol, along with a few drops of glacial acetic acid as a catalyst, was subjected to

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reflux for eight hours while being stirred [12]. After the reflux was completed, the resulting mixture was permitted to cool. The resulting solid was then filtered, dried, and recrystallized using chloroform.

3. Results and Discussion

The current study focused on the synthesis of a range of novel dimethylmaleimide heterocyclic derivatives.Synthesis of the new derivatives was based on 4-CN-dimethyl maleimidyl) phenyl hydrazide and the new compounds contain 1,2,4-triazole, 1, 3, 4-oxadiazole, different heterocycles including [10].

Dimethyl maleimide is a well known bioactive molety and the mentioned hetero cycles are also biologically active components this means combination of these two components in the newly synthesized compounds producing important bioactive new molecules leading to important applications. Synthesis of the chosen compounds was conducted through multiple processes, as outlined in Scheme (1). As indicated in scheme (1) different synthetic stratgies are followed in performing synthesis of the target compounds [10].

In the first step compound [1] ethyl-4-(N-dimethyl maleimidyl) benzoate was synthesized via reaction between ethyl-4-aminobenzoate and dimethyl maleic anhydride, in the second stage, compound [1] was subjected to a reaction with hydrazine hydrate, resulting in compound [2] via a nucleophilic substitution reaction 4-(N-dimethyl maleimidy1) phenyl hydrazide [10-12].

The FTIR spectra of chemical [1] displayed distinctive absorption bands at (1762) cm^{-1} and (1280, 1124)cm^{-1}, attributed to υ(C=O) ester and υ(C-O) ester, respectively. In contrast, these absorption bands were absent in the spectrum of compound [2] which instead displayed distinct absorption bands at(3236-3429) cm^{-1}, corresponding to υ(NH_2) and υ(NH). This point serves as compelling evidence of the successful production of hydrazide. The ^{1}H-NMR spectra of compound [1] exhibited signals at(δ=1.34) ppm and(1.95-2)ppm, pertaining to the protons of the CH_3 group and two CH_3 groups within the imide ring. Signals corresponding to $COCH_2$ protons and aromatic protons were observed at (δ=4.2-4.3) and (6.59-8.08) ppm, respectively [20].

Conversely ^{1}H-NMR spectrum of chemical [2] exhibited signals at (δ=1.91-2), (5.59-5.68), (6.51-7.59) and (9.1-9.28) ppm which are belong to protons of two CH_3 groups (NH_2), aromatic protons and (NH) proton respectively. It is noticable that the spectrum showed dis appearance of (OCH_2) signal and this is a secondary validation for the successful synthesis of hydrazide.The physical characteristics of chemicals [1] and [2] are presented in table (1), while their FTIR and ^{1}H-NMR spectral data are detailed in tables (6) and (11). Compound [2] serves as the crucial precursor from which the target compounds were synthesized. We are pleased to present The introduction of compound [2] in a condensation reaction with three aromatic aldehydes resulted in the formation of three new dimethyl maleimidyl Schiff bases [3-5, 12, 21].

The FTIR spectra of compounds [3-5] demonstrated absorption spectra at (1770-1782) cm^{-1}, (1689-1722) cm^{-1}, (1654-1691) cm^{-1}, (1602-1623) cm^{-1}, and (1377-1386) cm^{-1}, corresponding to asymmetrical (C=O) imide, symmetrical υ (C=O) imide, υ(C=O) amide, υ(C=N), and υ(C-N) imide, respectively. Conversely, the ^{1}H-NMR spectra of chemical [4] exhibit signals at (δ=1.99-2.16) and (6.65-8.3) ppm, essential to the protons of two (CH_3) groups and aromatic protons. Additional signals are observed at (δ=8.64-8.65) and (8.9-11.42) ppm,attributed to the (N=CH-) proton and the (NH) proton, respectively. The physical characteristics of the chemicals are presented in table (2), while their FTIR and ^{1}H-NMR spectral information are presented in tables (7) and (11).

Compounds [3-5] serve as the initial materials in the synthesis of the target. In the fourth step, compounds [3-5] are reacted with anthranilic acid to yield compounds [6-8] which are dimethyl maleimides featuring a dihydroquinazoline cycle. The FTIR spectra of chemicals [6-8] exhibited absorption bands at (1710-1720) cm^{-1}, (1620-1681) cm^{-1}, and (1361-1373) cm-1, corresponding to υ(C=O) imide, υ(C=O) amide, and υ(C-N) imide, this order [12].

^{1}H-NMR spectrum of chemical [7] showed signals at(δ= 1.99), (3.05) and (3.60-3-81) ppm which are belong to protons of two CH_3, (CH-N-) proton and (OCH_3) protons while other signals appeared at

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(δ=4.9-5.2), (6.5-8.3) and (8.63-8.84) ppm which correspond to (NH) protons, aromatic protons, and (NH) amide protons, respectively [9, 22]. The compounds' physical characteristics are displayed in table (3), and their FTIR and ^{1}H-NMR spectrum data are outlined in tables (8) and (11).

Compounds [3-5] are also utilized in the synthesis of additional target compounds [9-11] by introducing them into a reaction with acetic anhydride under reflux, resulting in the formation of new dimethyl maleimides that incorporate a 1,3,4-oxadiazole cycle [9-11]. The FTIR spectra of the chemicals [9-11] exhibited absorption bands at (1772)cm^{-1}, along with bands in the ranges of (1681-1733) cm^{-1}, (1654-1670) cm^{-1}, (1602-1627) cm^{-1}, and (1369-1371)cm^{-1}. The bands translate to the asymmetric (C=O) imide, symmetric υ(C=O) imide, υ(C=O) amide, (C=N), and (C-N) imide functionalities, this order [23].

The ^{1}H-NMR spectra of chemical [9] displayed signals at (δ=1.99 and 2.20)ppm, indicative of the protons from two (CH_3) groups and (CH_3-C) protons. Furthermore, signals at (δ=3.75)and within the range of (6.22-8.2)ppm were ascribed to the proton in the heterocyclic ring and the aromatic protons. The compounds' physical parameters are displayed in table 4, and their FTIR and ^{1}H-NMR spectrum data are outlined in tables (9) and (11).
The procedure entailed the synthesis of a new dimethyl morleimide [12] which integrates a 1,2,4-triazole ring through the reaction of compound [2] with CS2 in an alkaline environment, subsequently treated with hydrazine hydrate [11, 24] .

The FTIR spectra of chemical [12] exhibited absorption spectra at (1716 , 1701) cm^{-1}, (1629) cm^{-1}, and (1377) cm^{-1}, corresponding to asymmetric υ (C=O) imide, symmetric υ (C=O) imide, υ(C=N), and υ (C-N) imide, this order .The ^{1}H-NMR spectrum of this compound [12] displayed signals at (δ = 1.95-2.0), (7.45), and (8.4) ppm, attributed to the protons of two CH_3 groups, (NH2) protons, and aromatic protons, respectively [25].

Ultimately, molecule [2] was synthesized through a condensation reaction with the heterocyclic aldehyde pyrrole-2-carbaldehyde, yielding compound [13] dimethylmaleimide, which incorporates a pyrrole ring. The FTIR spectra of chemical [13] absorption bands were observed at (1778) cm^{-1}, (1706) cm^{-1}, (1625) cm^{-1}, (1610) cm^{-1}, and (1334) cm^{-1}, that correspond to asymmetrical υ(C=O) imide, symmetrical υ(C=O) imide, υ(C=O) amide, υ(C=N), and υ(C-N) imide, this order . The ^{1}H-NMR spectra of chemical [13] exhibited signals at (δ = 2.0-2.13)ppm and (6.13)ppm, corresponding to the protons of two (CH_3) groups and the (NH) amine proton. Additionally, signals at (δ = 6.44-8.3) ppm, (9.8)ppm, and (10.8)ppm were ascribed to aromatic protons, the (-N=CH-) proton, and the (NH) amide proton, respectively. The physical parameters of compounds Yassen and AL-Azzawi [12] and Jaber [13] are presented in Table (5), while their FTIR and ^{1}H-NMR spectrum data are detailed in Tables (10) and (11), respectively [12].

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Table 1.
Shows the physical characteristics of the chemicals [1, 2].

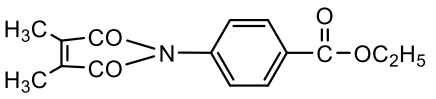
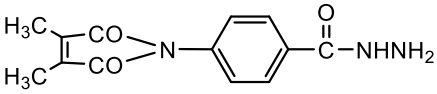
Comp. No.	Compound Structure	Colour	Yield %	Melting point °C	Recryst. Solvent
1		Off white	90	269-272	Ethanol
2		white	88	254-256	Dioxane

Table 2.
Shows the physical characteristics of the compounds [3-5].

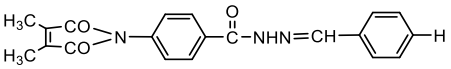
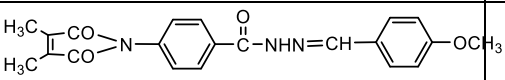
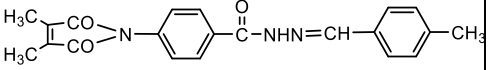
Comp. No.	Compound Structure	Colour	Yield %	Melting point °C	Recryst. Solvent
3		Off white	90	234-236	Chloroform
4		brown	88	248-250	Ethanol
5		bige	70	278-280	Chloroform

Table 3.
Shows the physical characteristics of the chemicals [6-8].

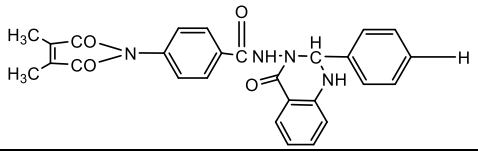
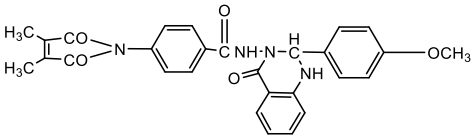
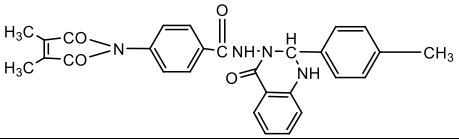
Comp. No.	Compound Structure	Colour	Yield %	Melting point °C	Recryst. Solvent
6		brown	80	>300	Hexan
7		Drak brown	76	>300	Dioxan
8		brown	68	>300	Hexan

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Table 4.
Shows the physical characteristics of the chemicals [9-11].

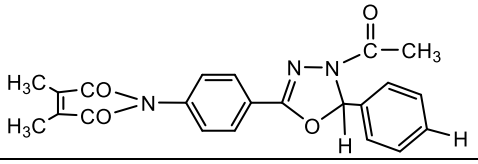
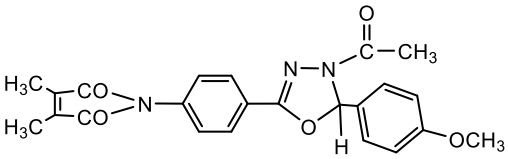
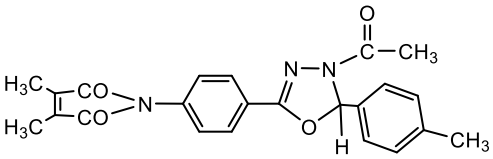
Comp. No.	Compound Structure	Colour	Yield %	Melting point ^{o}C	Recryst. Solvent
9		Yellow light	59	>300	Acetone
10		brown	54	290-292	Ethanol
11		brown	60	244-246	Acetone

Table 5.
Shows the physical characteristics of the chemicals [12, 13].

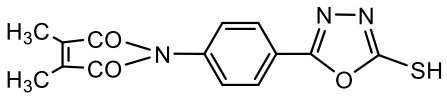
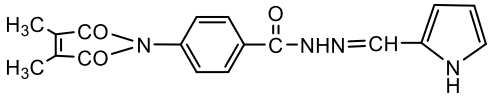
Comp. No.	Compound Structure	Colour	Yield %	Melting point ^{o}C	Recryst. Solvent
12		Off white	55	218-220	Dioxane
13		Yellow	95	284-286	Chloro form

Table 6.
Shows FTIR spectra (cm^{-1}) of chemicals [1, 2].

Comp. No.	υ (N-H , NH_2)	υ (C-H) Aromatic	υ (C-H) Aliphatic	υ (C=O) Ester	υ (C=O) Imide	υ (C=O) Amide	υ (C=C)	υ (C-N) Imide	υ (C-O) Ester
1	_____	3068	2983 2898	1762	1791 1683	_____	1598	1392	1280 1124
2	3429 3346 3305 3234	3031	2977 2879	_____	1670	1625	1604 1560	1350	_____

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Table 7.
Shows FTIR spectra (cm^{-1}) of chemicals [3-5].

Comp. No.	υ (N-H)	υ (C-H) Aromatic	υ (C-H) Aliphatic	υ (C=N)	υ (C=O) Imide	υ (C=O) Amide	υ (C=C)	υ (C-N) Imide	others
3	3203 3151	3055 3029	2952 2839	1623	1770 1689	1654	1556	1379	____
4	3153	3080	2977 2904 2877	1602	1782 1704	1691	1512	1386	**υ (C-O) ether** 1166 1026
5	3321 3145	3020	2960 2931 2875	1618	1772 1722	1658	1560	1377	_____

Table 8.
Shows FTIR spectra (cm^{-1}) of chemicals [6-8].

Comp. No.	υ (N-H)	υ (C-H) Aromatic	υ (C-H) Aliphatic	υ (C=O) Imide	υ (C=O) Amide	υ (C=C)	υ (C-N) Imide	others
6	3471 3371 3224	3058	2933 2860	1770 1718	1654 1620	1595	1361	____
7	3407 3355 3224	3020	2937 2881 2840	1710	1681	1604 1512	1373	**υ (C-O) ether** 1168 1027
8	3440 3402 3234	3029	2920 2852	1720	1670 1623	1608 1512	1367	_____

Table 9.
Shows FTIR spectra (cm^{-1}) of chemicals [9-11].

Comp.No.	υ (C-H) Aromatic	υ (C-H) Aliphatic	υ (C=O) Imide	υ (C=O) Amide	υ (C=N)	υ (C=C)	υ (C-N) Imide	others
9	3064	2979 2933 2881	1772 1712	1658	1627	1606 1552	1371	____
10	3068 3006	2970 2840	1772 1681	1654	1602	1577 1512	1369	**υ (C-O) ether** 1163 1024
11	3040	2927 2856 2817	1733	1670	1620	1589	1371	_____

Table 10.
Shows FTIR spectra (cm^{-1}) of chemicals [12, 13].

Comp. No.	υ (N-H , NH_2)	υ (C-H) Aromatic	υ (C-H) Aliphatic	υ (C=O) Imide	υ (C=O) Amide	υ (C=N)	υ (C=C)	υ (C-N) Imide
12	3407 3377 3346 3236	3060	2964 2929 2860	1716 1701	_____	1629	1606 1587	1377
13	3350 3301 3218	3068	2979 2856	1778 1708	1625	1610	1558	1334

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Table 11.
Shows ^{1}HNMR spectral data (ppm) of the prepared chemicals.

Comp. No.	(ppm) δ
1	δ = 1.34 (CH_3 protons) , δ=1.95-2.02 (protons of two CH_3 in imide ring), δ=4.2-4.3 (OCH_2), δ=6.59-8.08 (aromatic protons)
2	δ=1.95-2.02 (protons of two CH_3),δ=5.59-5.68 (NH_2 protons), δ=6.51-7.59 (aromatic protons) , δ=9.1-9.28 (NH proton)
4	, δ = 3.83-3.86 (OCH_3 proton))protons of two CH_3(δ = 2.0-2.16 aromatic protons) , δ = 8.64(-N=CH-) , δ = 8.9 (NH) (δ = 6.67-8. 0
5	δ=1.99-2.0 (protons of two CH_3),δ = 2.34 (CH_3 protons) , δ=6.65-8.3 (aromatic protons) , δ = 8.65 (-N=CH-), δ=11.42(NH proton)
7	δ=1.99 (protons of two CH_3),δ = 3.05 (-CH-N-,) δ = 3.6-3.81(OCH3 protons) , δ = 4.9-5.2 (NH proton) , δ=6.6-8.3 (aromatic protons) , δ=8.63-8.84 (NH amide)
9	δ=1.99 (protons of two CH_3),δ = 2.2(CH_3 -CO) ,δ = 3.37 (proton in hetro ring), δ=6.22-8.2 (aromatic protons
12	, δ = 7.45 (NH_2 proton))protons of two CH_3(δ = 1.95-2.0 aromatic protons)(δ = 8.40
13	= 2.0- 2.13 (proton of two CH3) δ = 6.13 (NH amine) δ = 6.44 – 8.3 δ (vinylic and aromatic protons) , δ = 9.8 (-N=CH-) ,δ = 10.8 (NH amide)

4. Examination of Biological Activity

The cup plate method was utilized to investigate the antibacterial and antifungal properties of the synthesized chemicals against four bacterial strains: Escherichia coli, Staphylococcus aureus, Streptococcus pyogenes, and Klebsiella pneumoniae, in addition to against the fungus Candida albicans, using Amoxicillin and Fluconazole as reference agents, with DMSO serving as the sample solution [23]. The zones of inhibition generated by the substances under investigation were quantified, and the outcomes are detailed in table 12.

Table 12.
Inhibition zones of chemicals [6-13].

Comp.no	Staphylococcus aureus	Staphylococcus pyogenes	Escherichia Coli	Klebsiella pneumoniae	Candida albicans
6	14	12	-	-	19
7	14	12	-	-	18
8	12	13	-	-	29
9	18	17	17	20	21
10	14	17	17	17	23
11	26	26	23	24	34
12	24	27	-	-	43
13	17	14	18	15	25
Amoxicillin	15	18	17	15	0
Fluconazole	0	0	0	0	19

Table 12 demonstrates that compounds [11, 12] had significant action against Staphylococcus aureus and Streptococcus. Compounds [6, 7, 9, 10, 13] derived from pyogenes bacteria shown significant efficacy against Staphylococcus aureus, while compounds [9, 10] demonstrated pronounced activity against Streptococcus pyogenes. Compounds [6-8, 13] displayed moderate activity against Streptococcus pyogenes, and compound [11] revealed exceptional action against Escherichia coli. Compounds [9, 10, 13] had significant action, whereas compounds [6-8, 12] demonstrated no activity against this bacterium (17). Compounds [9, 11] had significant efficacy against Klebsiella pneumoniae, while compounds [10, 13] shown considerable activity. Compounds [6-8, 12] shown no action against this bacterium. Conversely, substances [8, 11, 12] had significant efficacy against Candida fungus, whilst other examined compounds shown considerable effectiveness against this fungi.

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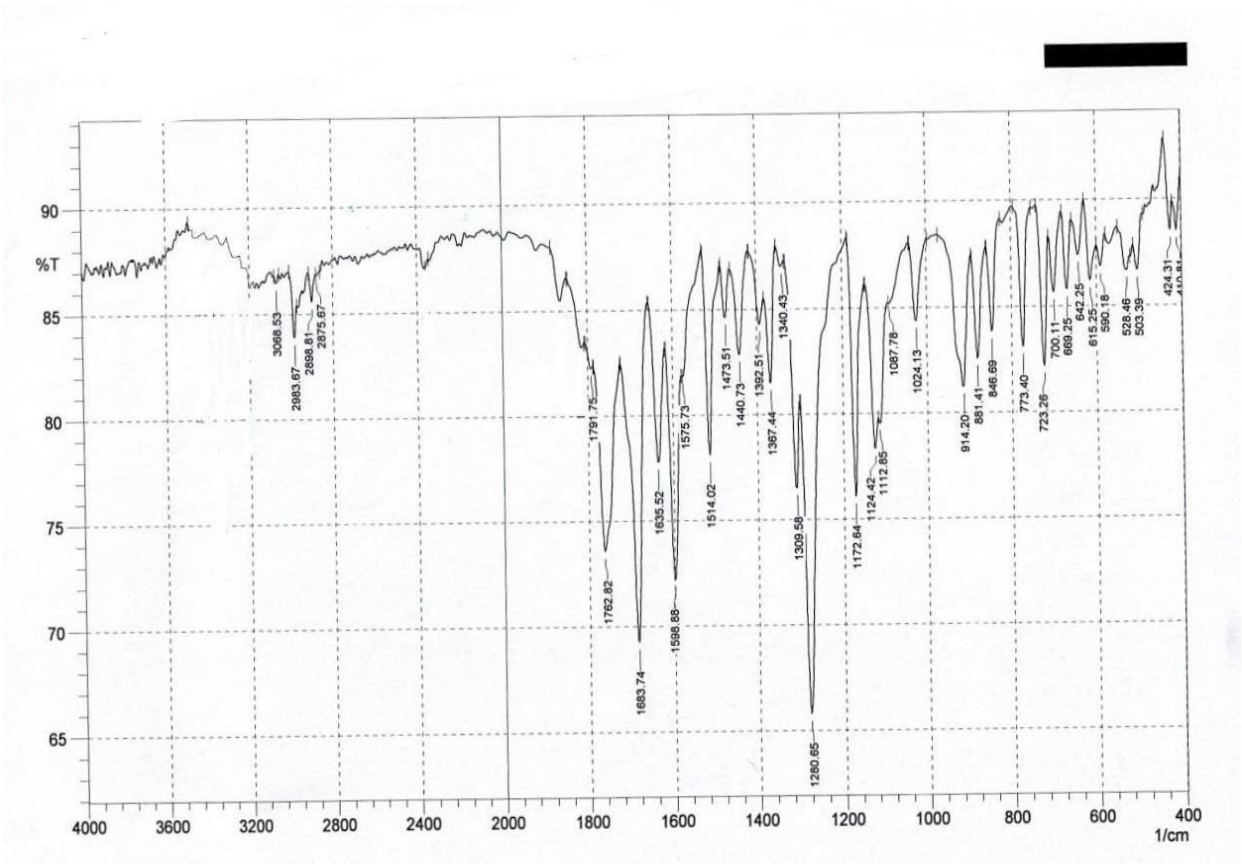

Figure 1.
FTIR spectrum of of chemical [1].

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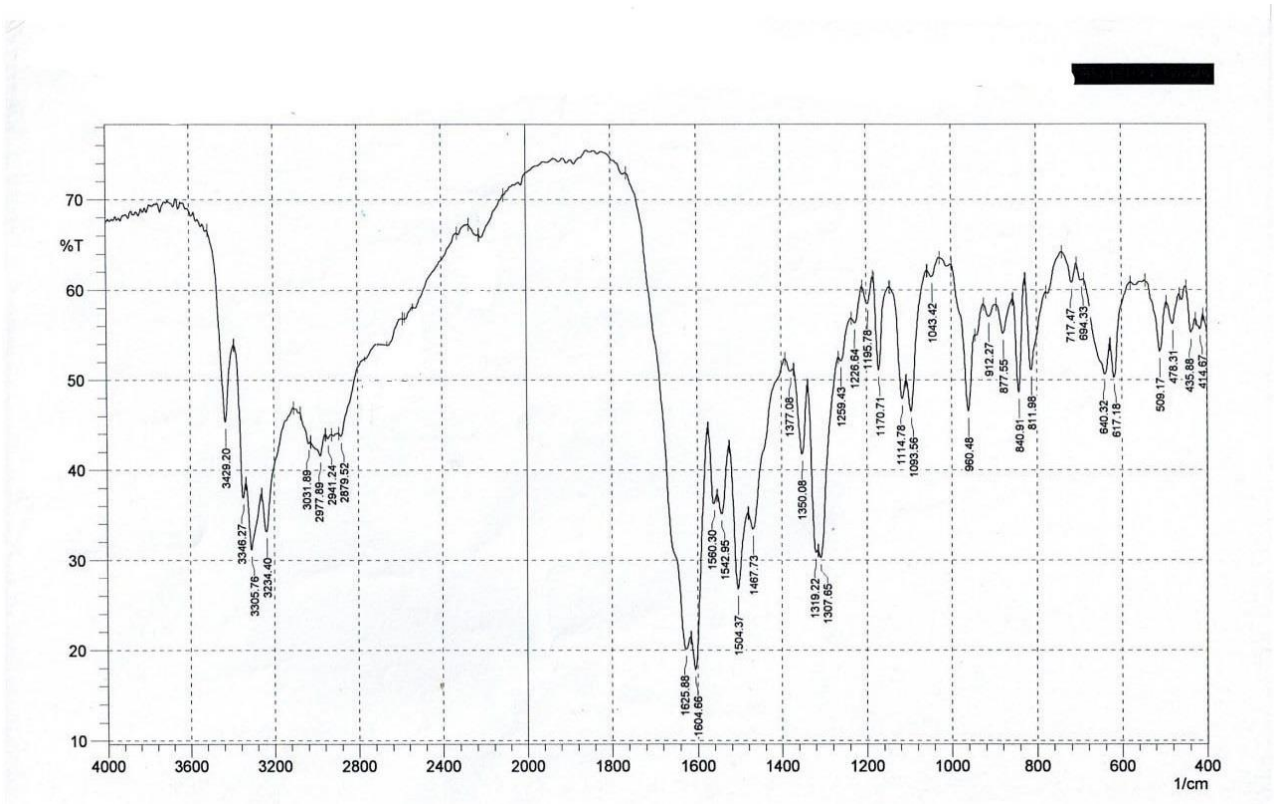

Figure 2.
FTIR spectrum of of chemical [2].

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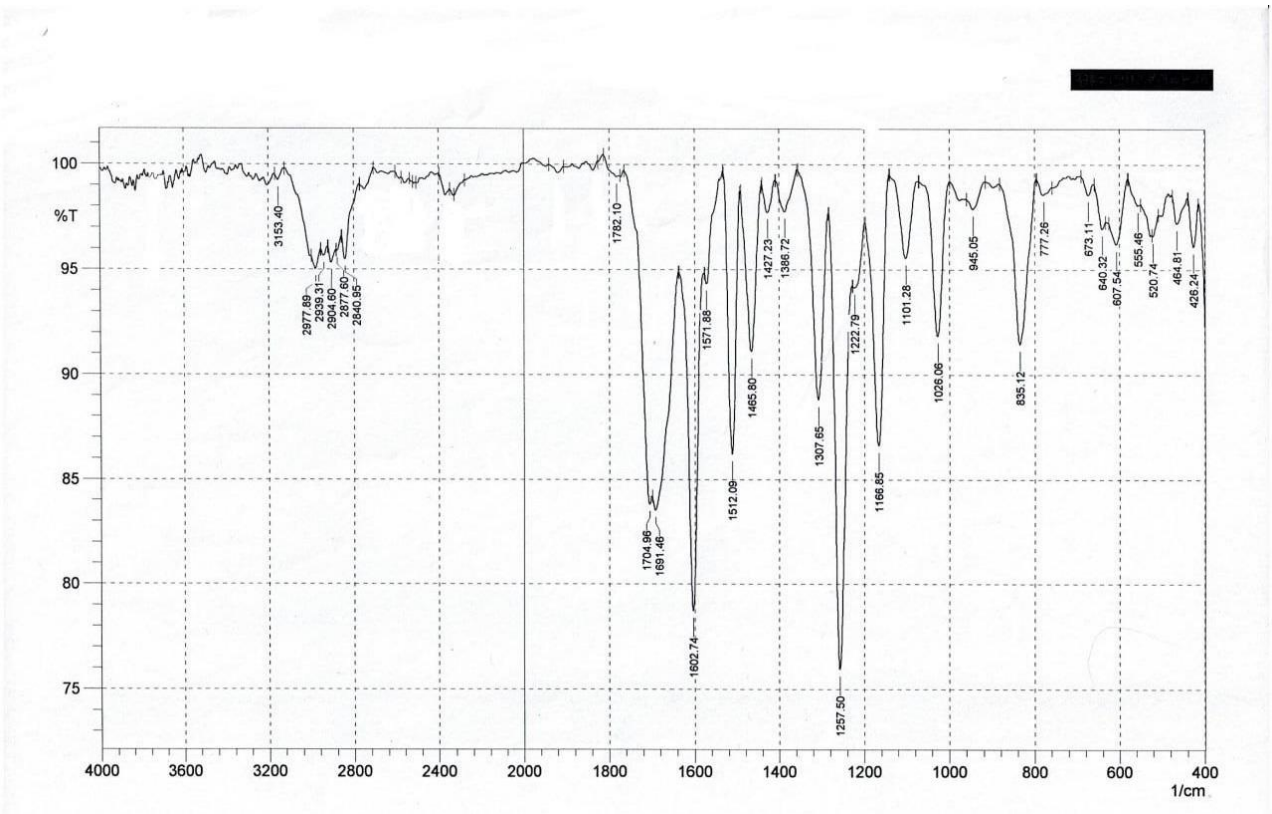

Figure 3.
FTIR spectrum of of chemical [4].

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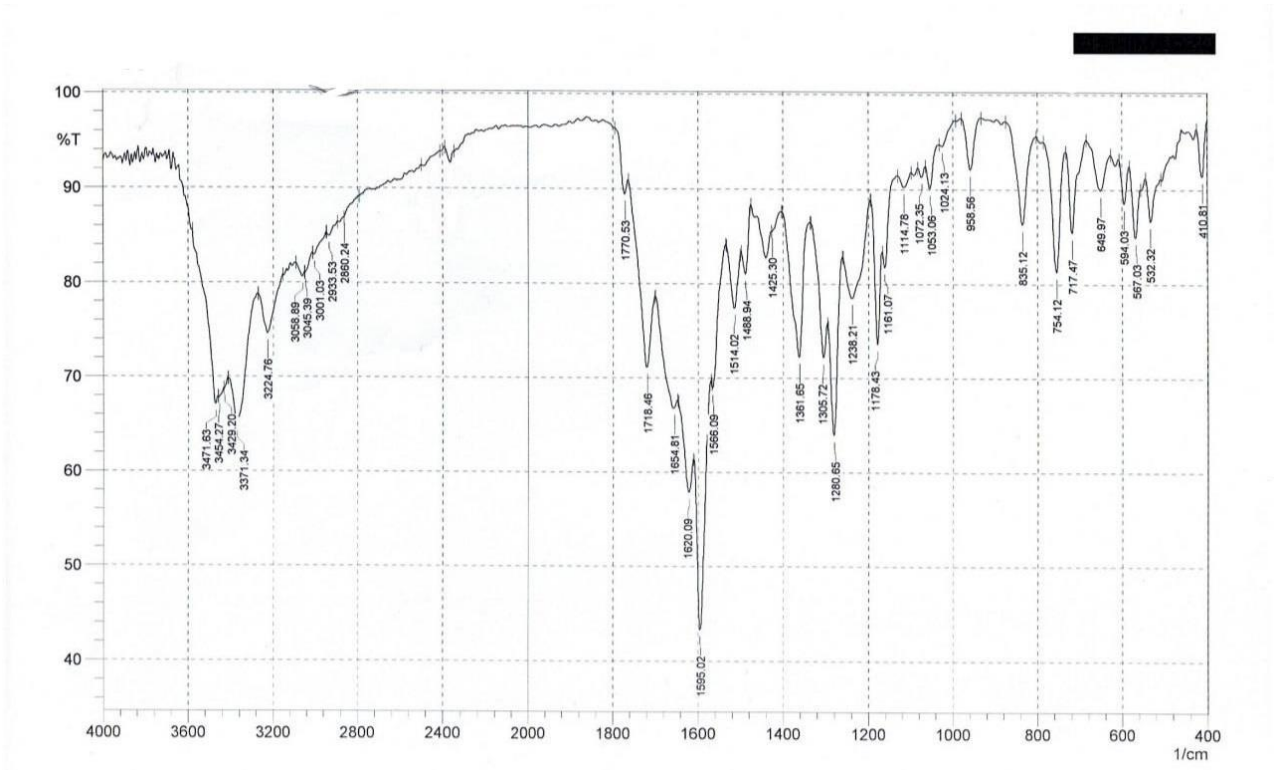

Figure 4.
FTIR spectrum of of chemical [6].

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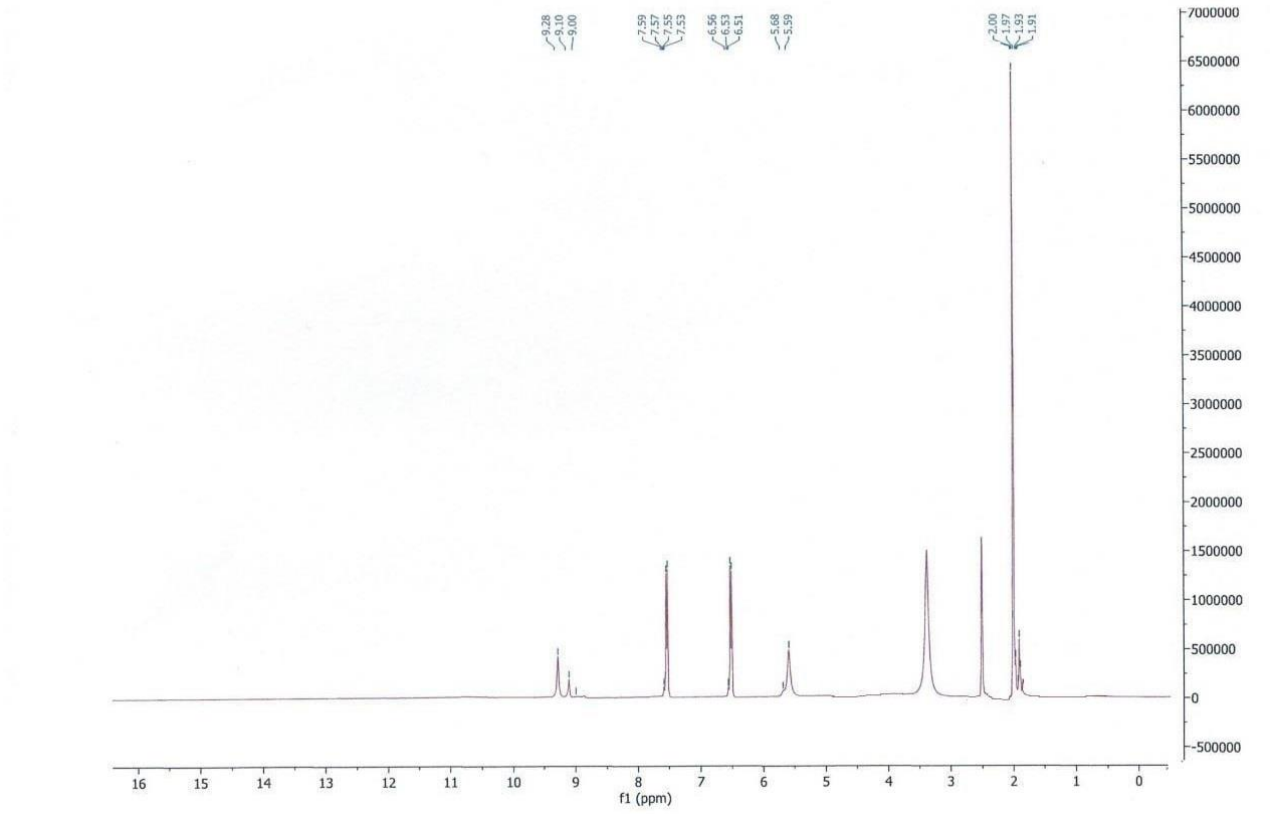

Figure 5.
^{1}H-NMR spectrum of of chemical [2].

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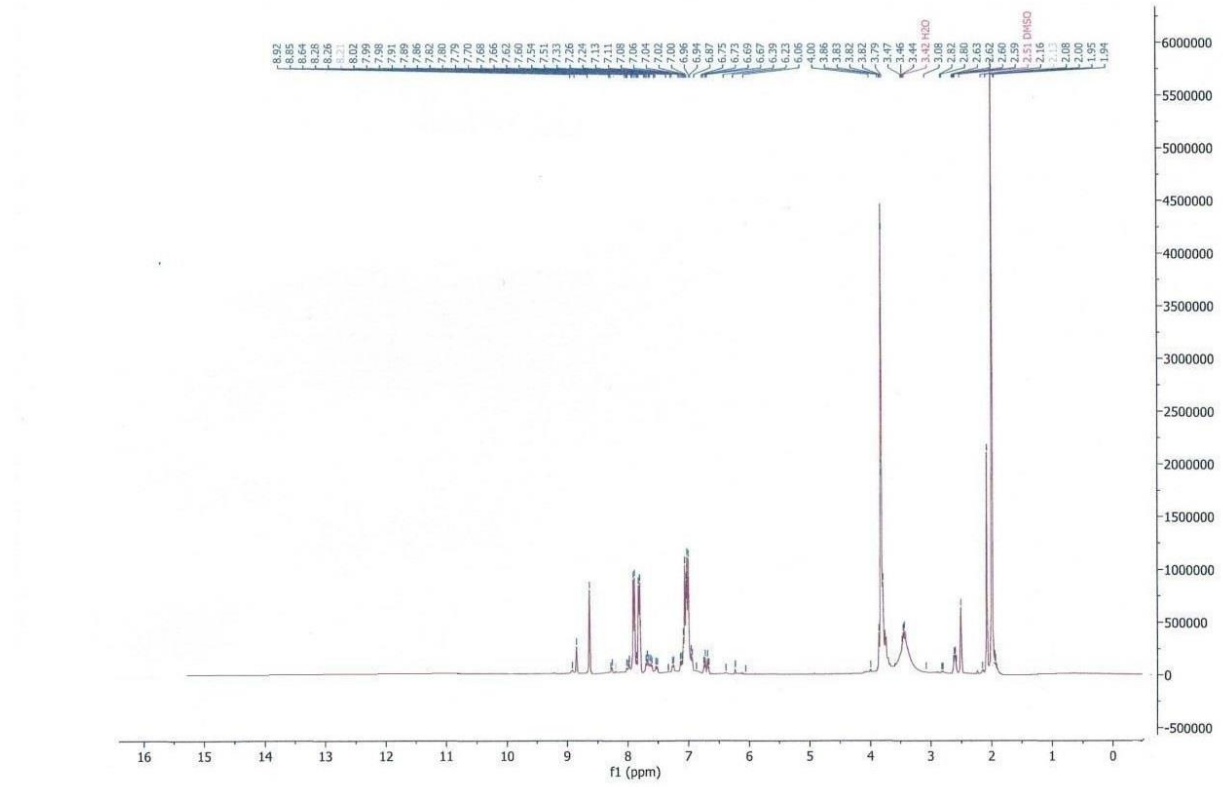

Figure 6.
^{1}H-NMR spectrum of chemical [4].

Transparency:

The authors confirm that the manuscript is an honest, accurate, and transparent account of the study; that no vital features of the study have been omitted; and that any discrepancies from the study as planned have been explained. This study followed all ethical practices during writing.

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