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Design, Synthesis and Antimicrobial Activity Screening of Novel Cyclic Imides Derived From Heterocyclic Imines

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Abstract

In this work, nine new cyclic imides **10-18** were synthesized starting with 4-aminoacetophenone and heterocyclic amines (2-aminothiazole, 4-aminopyridine, and 4-aminoantipyrine). The first step included a condensation between 4-aminoacetophenone and heterocyclic amines to afford the corresponding imines **1-3** in 82-90% yields. These imines (**1-3**) were then treated with phthalic and maleic anhydrides to give heterocyclic iminophthalamic and maleamic acids **4-9**, which were dehydrated using fusion conditions to provide the desired phthalimides and maleimides (**10-15**) in yields ranging from 75 to 92%. Heterocyclic imino tetrachlorophthalimides **16-18** were synthesized (83-88% yields) in a one-pot procedure *via* the reaction of imines **1-3** with tetrachlorophthalic anhydride in the presence of glacial acetic acid as a catalyst. Results indicated that the prepared cyclic imides (**1-3**) exhibit good antimicrobial activity.

Keywords: Antimicrobial activity, Cyclic imides, Heterocyclic amines.

تصميم، تحضير و تقدير الفعالية المضادة للميكروبات لايمايدات حلقيه جديدة مشتقه من ايمينات حلقيه غير متجانسة

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الخلاصة

في هذا العمل، تم تحضير تسعة إيمايدات حلقيه جديدة **10-18** بدءاً من 4-أمينوأسيتوفينون و الأيمينات الحلقيه غير المتجانسة (2-أمينو ثايازول، 4-أمينو بريدن، و 4-أمينو أنتي بايرين). تضمنت الخطوة الأولى تكاثفا بين 4-أمينوأسيتوفينون و الأيمينات الحلقيه غير المتجانسة مما اسفر عن تكوين الإيمينات المقابلة **1-3** بنسبة 82-90%. تم معاملة هذه الايمينات **1-3** لاحقاً مع انهيدريد الفثاليك و أنهيدريد الماليك منتجاً مركبات حوامض فثال أميك و حوامض مالي أميك ايمينو حلقيه غير متجانسه **4-9** و هذه بدورها تم سحب الماء منها بتطبيق عملية الصهر مما أسفر عن تكوين مركبات فثال ايماید و مالي ايماید مرغوبة **10-15** بنسبة 75-92%. تم تحضير مركبات رباعي كلورو فثال ايماید ايمينو حلقيه غير متجانس **16-18** بنسبة 83-88% عن طريق مسار واحد و ذلك بمفاعلة الايمينات **1-3** مع رباعي كلورو انهيدريد الفثاليك بوجود حامض الخليك الثلجي كعامل مساعد. أشارت النتائج إلى أن الإيمايدات الحلقيه المحضرة **1-3** تظهر نشاطاً جيداً مضاداً للميكروبات.

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1. Introduction

Heterocyclic compounds are still the most significant class of organic molecules, playing a crucial role in the production of medications and pharmaceuticals. In addition to their great importance in various fields, including agriculture, industry, and medicine, they exhibit various biological activities like antimicrobial, antioxidant, anti-inflammatory, antibacterial, analgesic, and anticancer [1-6]. On the other hand, cyclic imides are well known important building segments for the synthesis of pharmaceuticals, composites, resins, drugs, and a huge number of different polymers [7,8]. The compounds display various pharmacological and biological features, such as antimicrobial, analgesic, anticancer, and antibacterial effects [9-12]. Moreover, Schiff base compounds have gained great importance as a result of their wide spectrum of biological properties and applications like anti-corrosion, dyes, and ligands in coordination chemistry [13-15]. Schiff bases are the results of the condensation reaction of primary amines with carbonyl compounds, and the presence of an imine group in their structures exhibits them important activities, including, antimicrobial, antioxidant, anti-inflammatory, antiviral, and antibacterial [12,15,16] leading to various applications. In light of all these findings, it appears worthwhile to produce new molecules that combine these three active components (cyclic imide, heterocycle, and Schiff base) in the same molecule, leading to new compounds with interesting biological activities. In this work, new cyclic imides bearing Schiff bases and heterocyclic moieties will be prepared by a three-step procedure. The prepared products could be subjected to antimicrobial tests to evaluate their activities.

2. Experimental part

2.1. Materials and instrumentation

All chemicals were obtained from commercial sources unless specified otherwise. The melting points were measured using a Thomas Hoover in open capillary tubes and are uncorrected. A Shimadzu 8400 FT-IR spectrometer was used to record infrared spectral data in 500-4000 cm^{-1} region. Spectral data for ^1H NMR and ^{13}C NMR were captured using a Bruker AV400 spectrometer. Chemical shifts are expressed in ppm downfield from tetramethylsilane (TMS) as an internal standard or deuterated DMSO in ^1H NMR as a reference.

2.2. Synthesis

2.1. *N*-(4(*N*-heterocycles))-methyl-4-aminobenzylidene **1-3**

A solution of 4-aminoacetophenone (1.35 g, 10 mmol), heterocyclic amines (2-aminothiazole, 4-aminopyridine, and 4-aminoantipyrine) (10 mmol), and a few drops of glacial acetic acid in ethanol absolute (25 mL) was refluxed for 8 hours [17]. After completing the reflux, the reaction was cooled in an ice bath. The solid crude material was filtered and recrystallized using a suitable solvent to give the desired products **1-3**. The physical properties of these products are shown in Table 1.

2.2. *N*-(4(*N*-heterocycles))-methyl benzylidene phthalamic and maleamic acids **4-9**

Compounds **1-3** (10 mmol) in acetone (20 mL) gradually added to a solution of phthalic or maleic anhydride (10 mmol) in acetone (15 mL) and stirred for 2 hours at 0 °C [18]. The solid crude material was filtered and recrystallized using a suitable solvent to afford the desired products **4-9**. The physical properties of these products are shown in Table 2.

2.3. *N*-(4(*N*-heterocycles))-methyl benzylidene phthalimide and maleimide **10-15**

Phthalamic and maleamic acids **4-9** were heated to fusion in a sand bath. After two hours of fusion [18], the mixture was then allowed to cool to room temperature. The solid crude

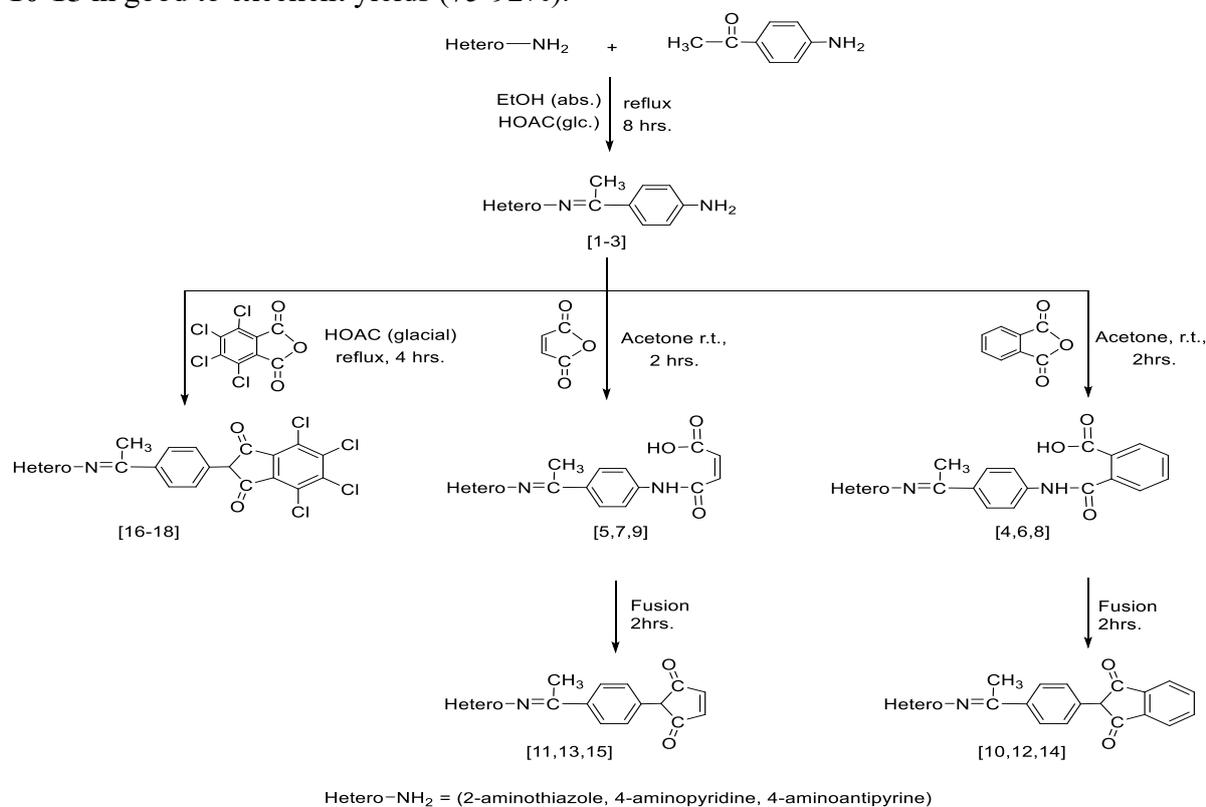
material was then filtrated and recrystallized to provide the desired products **10-15**. The physical properties of these products are listed in Table 3.

2.4. *N*-(4(*N*-heterocycles))-methyl benzylidene tetrachlorophthalimide **16-18**

A mixture of compounds **1-3** (10 mmol), tetrachlorophthalic anhydride (2.86 g, 10 mmol), and glacial acetic acid (20 mL) was refluxed for 4 hours [19]. When the reflux had reached completion, the mixture was added to ice water and thoroughly stirred. The precipitate that was formed was collected by filtering, washed with diethyl ether, dried, and recrystallized using an appropriate solvent.

3. Results and discussion

Schiff bases, heterocycles, and cyclic imides are widely recognized by researchers as biologically active entities that exhibit a variety of biological activities. We synthesized novel compounds by integrating these three active components within a single molecule, as this combination has the potential to generate compounds with intriguing biological activities. The target compounds (**10-18**) were synthesized using a number of steps, as illustrated in Scheme 1. In the first step, three amino heterocyclic Schiff bases (**1-3**) were synthesized *via* the condensation reaction of three heterocyclic amines (2-aminothiazole, 4-aminopyridine, and 4-aminoantipyrine) with 4-aminoacetophenone using ethanol absolute as a solvent and glacial acetic acid as a catalyst. Due to the amine group present in the Schiff bases **1-3**, there was an opportunity to functionalize them in the second step by reacting with two cyclic anhydrides (phthalic and maleic) to give the corresponding heterocyclic iminophthalamic and maleamic acids **4-9**. The synthesized amic acids **4-9** were then dehydrated by the fusion process in the third step to afford the target heterocyclic iminophthalimides and maleimides **10-15** in good to excellent yields (75-92%).



Scheme 1 : Synthesis of heterocyclic imidyl Schiff bases

Furthermore, three heterocyclic iminotetrachlorophthalimides **16-18** were synthesized in 82-90% yields *via* a one-pot reaction of compounds **1-3** with tetrachlorophthalic anhydride and

glacial acetic acid. At the first stage of this reaction, the formation of amic acids occurs, which are then directly dehydrated to cyclic imides [19]. The physical properties of Schiff bases **1-3**, amic acids **4-9** and cyclic imides **10-18** are listed in Tables 1, 2, and 3, respectively. The chemical structures of the compounds synthesized in this study were verified using spectral data from FT-IR, ^1H NMR, and ^{13}C NMR. The FTIR spectra of amino heterocyclic Schiff bases **1-3** showed clear absorption bands at $(3460-3226)\text{ cm}^{-1}$ due to symmetric and asymmetric $\nu(\text{NH}_2)$. The spectra also showed absorption bands at $(1649-1641)\text{ cm}^{-1}$ and others at $(1593-1562)\text{ cm}^{-1}$, which belong to $\nu(\text{C}=\text{N})$ and $\nu(\text{C}=\text{C})$, respectively [20]. The FTIR spectra amic acids **4-9** showed absorption bands at $(3440-3180)\text{ cm}^{-1}$ due to $\nu(\text{N-H})$ amide group and $\nu(\text{O-H})$ carboxyl group.

Furthermore, $\nu(\text{C}=\text{O})$ carboxyl and $\nu(\text{C}=\text{O})$ amide absorption bands appeared at $(1720-1703)\text{ cm}^{-1}$ and $(1685-1649)\text{ cm}^{-1}$, respectively. While the absorption bands at $(1595-1506)\text{ cm}^{-1}$ and $(1649-1631)\text{ cm}^{-1}$ are for $\nu(\text{C}=\text{C})$ and $\nu(\text{C}=\text{N})$, respectively [20]. The FTIR spectral data of cyclic imides **10-18** indicated disappearance of the stretching absorption for the (O-H) carboxyl, $\nu(\text{N-H})$ amide, $\nu(\text{C}=\text{C})$ carboxyl and $\nu(\text{C}=\text{O})$ amide and appearance of new absorption bands at $(1789-1743)\text{ cm}^{-1}$, $(1733-1714)\text{ cm}^{-1}$ and $(1390-1380)\text{ cm}^{-1}$, which are attributed to asymmetric $\nu(\text{C}=\text{O})$ imide, symmetric $\nu(\text{C}=\text{O})$ imide and $\nu(\text{C-N})$ imide respectively [18]. The FTIR spectral data of compounds **1**, **4** and **10** are displayed in Figures 1, 2 and 3, respectively. These are key evidences supporting the success of the dehydration reaction and cyclic imides formation. Tables 4, 5 and 6 show FTIR spectral data for compounds **1-3**, **4-9** and **10-18**, respectively. The synthesized compounds are additionally characterized using $^1\text{H-NMR}$ and $^{13}\text{C-NMR}$ spectroscopy. $^1\text{H-NMR}$ spectrum of N-thiazolyl Schiff base **1** showed singlet signals at (2.41) and (6.05) ppm which correspond to the CH_3 and NH_2 protons. The signals associated with aromatic and thiazole ring protons appeared at (6.64) ppm and (7.73-6.95) ppm, respectively [20]. In the $^{13}\text{C-NMR}$ spectrum of compound **1**, signals were showed at (26.28), (139.06-107.07) and (154.06, 169.41) ppm due to CH_3 , aromatic and thiazole ring and (C=N) carbon. The

$^1\text{H-NMR}$ spectral data of pyridine Schiff base **2** showed chemical shifts at (2.42), (6.10), and (8.04-6.51) ppm due to CH_3 , NH_2 and aromatic protons, respectively [20]. The $^{13}\text{C-NMR}$ spectral data of compound **2** showed signals (26.27), (149.81-109.41), (154.13 and 154.84) ppm, corresponding to methyl, aromatic, and (C=N) carbons. In contrast, the $^1\text{H-NMR}$ spectrum of thiazolyl imino phthalamic acid **4** showed signals at (2.11), (6.64-6.61) ppm belonging to CH_3 protons and thiazole ring protons, while the aromatic, (NH) amide and (OH) carboxyl proton show signals at (8.02-7.60), (10.75) and (13.17) ppm, respectively [20]. The $^{13}\text{C-NMR}$ spectrum of compound **4** showed signals at (26.92), (132.36-119.21) and (139.08, 144.38) ppm belong to CH_3 , thiazole ring, aromatic and (C=N) carbons, respectively, while signals at (167.83) and (168.45) ppm correspond to (C=O) amide and (C=O) carboxyl group. The ^1H NMR spectra of compounds thiazolyl imino phthalimide **10** and pyridyl imino phthalimide **12** showed chemical shifts at (2.65) and (8.77-6.56) ppm belonging to the CH_3 and aromatic protons, respectively. The $^{13}\text{C-NMR}$ spectral data of these compounds showed signals at (27.33-27.32), (140.01-121.11), (150.95-136.29) and (167.14-166.69) ppm due to the methyl, aromatic, C=N, and C=O carbons, respectively [9]. The ^1H NMR spectra of compounds pyridyl imino maleimide **13** and antipyryl imino maleimide **15** showed signals at 2.24-2.16, 7.0-5.02, and 8.18-6.80 ppm belonging to the CH_3 , thiazole ring, and aromatic protons, respectively. The ^1H NMR spectrum of compound **15** showed signals at (2.66) and (3.36) ppm for two methyl groups in the antipyryne ring protons. The ^{13}C NMR spectral data of compounds **13** and **15** showed signals at (35.63-26.48), (136.78-112.31), (152.5-152.11), and (176.16-160.1) ppm attributed to the methyl, thiazole ring, aromatic, C=N, and C=O (imide) carbons, respectively. In the ^{13}C NMR spectrum of compound **15**,

other signals appeared at (27.29), (51.85) and (174.24) ppm, which belong to the carbons of two methyl groups and C=O (amide) carbons at the antipyrine ring [20]. Finally, the ^1H NMR spectrum of compound 18 showed signals at (2.28), (2.66), (3.28) and (8.17-6.58) ppm, corresponding to the CH_3 , two CH_3 groups at the antipyrine ring, and aromatic protons, respectively. Figures 4-9 display the ^1H NMR and ^{13}C NMR spectra of compounds 1, 4, and 10, respectively. All details of the ^1H NMR and ^{13}C NMR spectral data of the produced compounds are listed in Tables 7 and 8.

Table 1 : Some physical properties of the prepared compounds 1-3

No.	Compound structure	Color	Yield (%)	m.p. (°C)	Recrystallization solvent
1		Off white	84	59-60	Benzene
2		Pale Yellow	90	77-79	Benzene
3		orange	82	60-62	Ethanol

Table 2 : Some physical properties of the prepared compounds 4-9

No.	Compound structure	Color	Yield (%)	m.p. (°C)	Recrystallization solvent
4		Off white	80	170-172	Ethanol
5		Brown	90	191-193	Ethanol
6		White	85	116-118	Ethanol
7		Yellow	83	200-202	Dioxane
8		Yellow	77	100-102	Acetone
9		Pale Yellow	75	182-184	Acetone

Table 3 : Some physical properties of the prepared compounds 10-18

No.	Compound structure	Color	Yield (%)	m.p. (°C)	Recrystallization solvent
10		Gray	92	240-242	Acetone
11		Black	90	286-288	Acetone
12		Brown	88	188-190	Ethanol
13		Pink	75	168-170	Ethanol
14		Pale brown	91	193-195	Ethanol
15		Brown	85	245-247	Acetone
16		Dark brown	87	198-200	Petroleum ether b.p. (40-60)
17		Black	83	280-282	Dioxane
18		Yellow	88	271-273	Ethanol

Table 4 : Characteristic FT-IR spectral data (ν , cm^{-1}) of compounds 1-3

No.	$\nu(\text{NH}_2)$ Amine	$\nu(\text{C-H})$ Aromatic	$\nu(\text{C-H})$ Aliphatic	$\nu(\text{C=N})$ Imine	$\nu(\text{C=C})$ Aromatic	Other bands
1	3400 3332 3226	3083	2952 2825	1649 1641	1591 1564	
2	3460 3396 3332	3037	2997 2854	1647	1593 1562	
3	3394 3332 3226	3064	2925 2875	1647	1589 1562	$\nu(\text{C=O})$ amide 1647 (overlap)

Table 5 : Characteristic FT-IR spectral data (ν , cm^{-1}) of compounds **4-9**

No.	$\nu(\text{O-H})$ $\nu(\text{N-H})$	$\nu(\text{C-H})$ Aromatic	$\nu(\text{C-H})$ Aliphatic	$\nu(\text{C=O})$ Carboxyl	$\nu(\text{C=O})$ Amide	$\nu(\text{C=N})$ Imine	$\nu(\text{C=C})$ Aromatic	Other bands
4	3431 3247 3180	3053	2937 2840	1716	1670	1649	1585 1527	
5	3271 3197	3082 3014	2930 2879	1712	1677	1633	1537 1506	
6	3431 3336 3290	3064	2977 2931 2870	1703	1685	1641	1589 1558	
7	3434 3338 3230	3068	2977 2880	1718	1680	1641	1595 1556	
8	3440 3427 3259	3062 3012	2931 2860	1720	1676	1631	1593 1562	$\nu(\text{C=O})$ Amide antipyrene 1649
9	3433 3280 3193	3008	2947 2883	1718	1649	1637	1593 1542	$\nu(\text{C=O})$ Amide antipyrene 1649 (overlap)

Table 6 : Characteristic FT-IR spectral data (ν , cm^{-1}) of compounds **10-18**

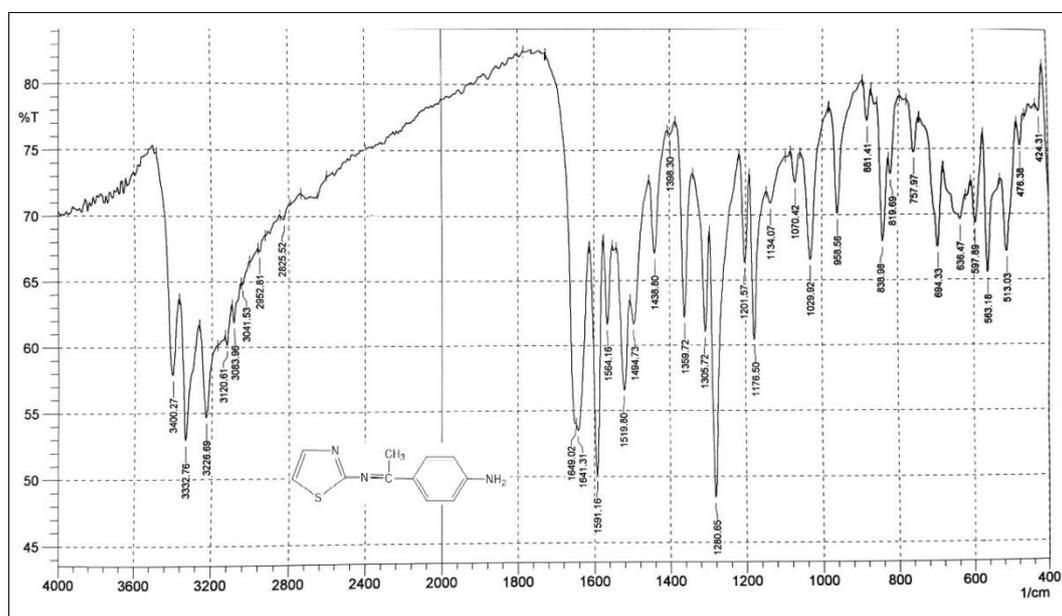
No.	$\nu(\text{C-H})$ Aromatic	$\nu(\text{C-H})$ Aliphatic	$\nu(\text{C=O})$ Imide (Asymmetric and symmetric)	$\nu(\text{C=N})$ Imine	$\nu(\text{C=C})$ Aromatic	$\nu(\text{C-N})$ Imide	Other bands
10	3058	2920 2819	1743 1714	1677 1629	1600 1512	1382	
11	3080	2972 2933 2850	1782 1714	1677 1629	1595	1380	
12	3099	2983 2894	1747 1716	1679 1649	1573	1384	
13	3074 3002	2925 2856	1776 1714	1685 1625	1598	1384	
14	3060	2929 2820	1785 1720	1633	1595	1380	$\nu(\text{C=O})$ Amide 1679
15	3066	2929 2819	1774 1720	1637	1595	1390	$\nu(\text{C=O})$ Amide 1677
16	3072	2921 2864	1784 1726	1683	1602 1575	1382	$\nu(\text{C-Cl})$ 1103
17	3055	2972 2935 2873	1778 1726	1683 1641	1595 1558	1384	$\nu(\text{C-Cl})$ 1101
18	3076 3045	2962 2927 2825	1789 1733	1635	1595	1388	$\nu(\text{C=O})$ Amide 1670 $\nu(\text{C-Cl})$ 1047

Table 7 : ^1H NMR spectral data (δ , ppm) of compounds **1,2,4,10,12,13,15**, and **18**

No.	^1H NMR spectral data (δ , ppm)
1	2.41 (3H, CH ₃), 6.05 (2H, NH ₂), 6.64-6.54 (2H, Thiazole ring), 7.73-6.95 (4H, Ar-H)
2	2.42 (3H, CH ₃), 6.10 (2H, NH ₂), 8.04-6.51 (8H, Ar-H)
4	2.11 (3H,CH ₃), 6.64-6.61 (2H, Thiazole ring), 8.02-7.60 (8H, Ar-H), 10.75 (1H, NH), 13.17 (1H, OH)
10	2.65 (3H, CH ₃), 8.14-7.65 (10H, Thiazole ring and Ar-H)
12	2.65 (3H, CH ₃), 8.77-6.56 (12H, Ar-H)
13	2.24-2.18 (3H, CH ₃), 7.0 (2H, Thiazole ring), 8.12-7.38 (8H, Ar-H).
15	2.16, 2.66 (6H, CH ₃), 3.36 (3H, N-CH ₃), 5.05-5.02 (2H, Thiazole ring), 8.18-6.80 (9H, Ar-H)
18	2.28, 2.66 (6H, CH ₃), 3.28 (3H, N-CH ₃), 8.17-6.58 (9H, Ar-H)

Table 8 : ^{13}C NMR spectral data (δ ,ppm) of compounds **1,2,4,10,12,13**, and **15**

No.	^{13}C NMR spectral data (δ , ppm)
1	26.28 (CH ₃), 139.06-107.07 (Thiazole ring and Ar-C) 154.06, 169.41 (C=N)
2	26.27(CH ₃), 149.81-109.41 (Ar-C), 154.13, 154.84 (C=N)
4	26.92 (CH ₃), 132.36-119.21 (Ar-C), 139.08, 144.38 (C=N), 167.83 (C=O) amide, 168.45 (C=O) carboxyl
10	27.33 (CH ₃), 135.34-124.04 (Thiazole ring and Ar-C), 136.29, 136.51 (C=N), 167.14 (C=O) imide
12	27.32 (CH ₃), 140.01-121.11 (Ar-C), 150.95 (C=N), 167.13-166.69 (C=O) imide
13	35.63 (CH ₃), 134.0 -124.97 (Thiazole ring and Ar-C), 152.5 (C=N), 160.1 (C=O) imide
15	26.48, 27.29 (CH ₃),51.85 (N-CH ₃), 136.78-112.31 (Thiazole ring and Ar-C), 152.11(C=N), 174.24 (C=O) amide, 176.16 (C=O) imide

**Figure 1 :** FT-IR spectrum of compound **1**

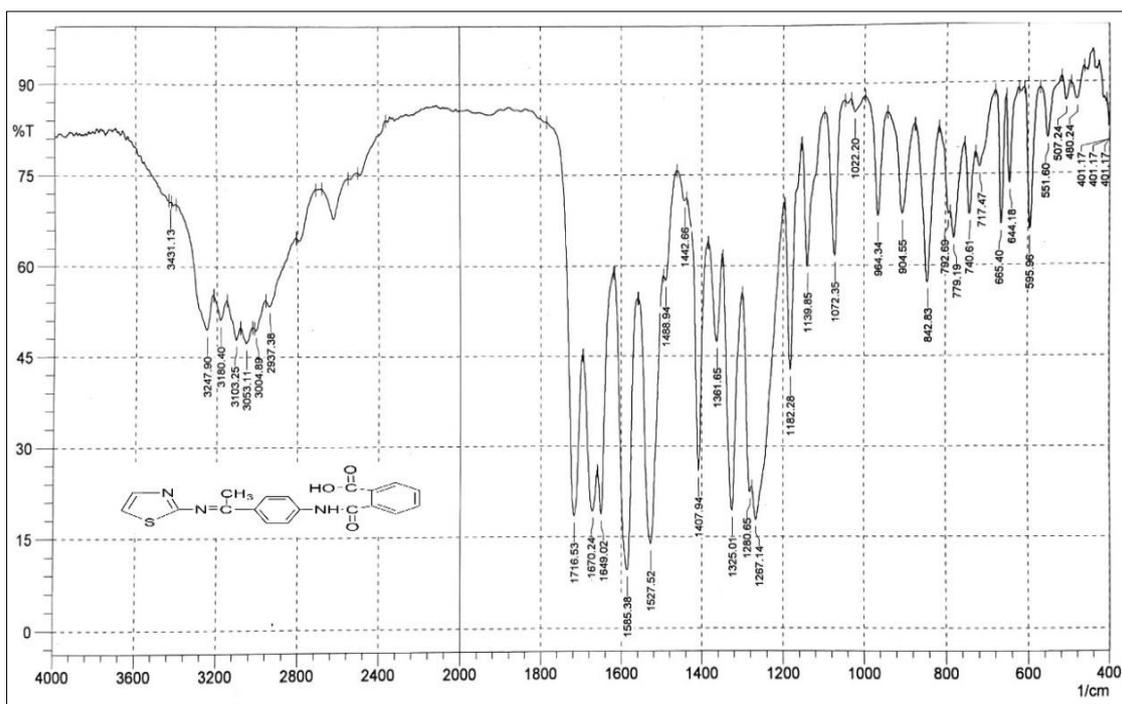


Figure 2 : FT-IR spectrum of compound 4

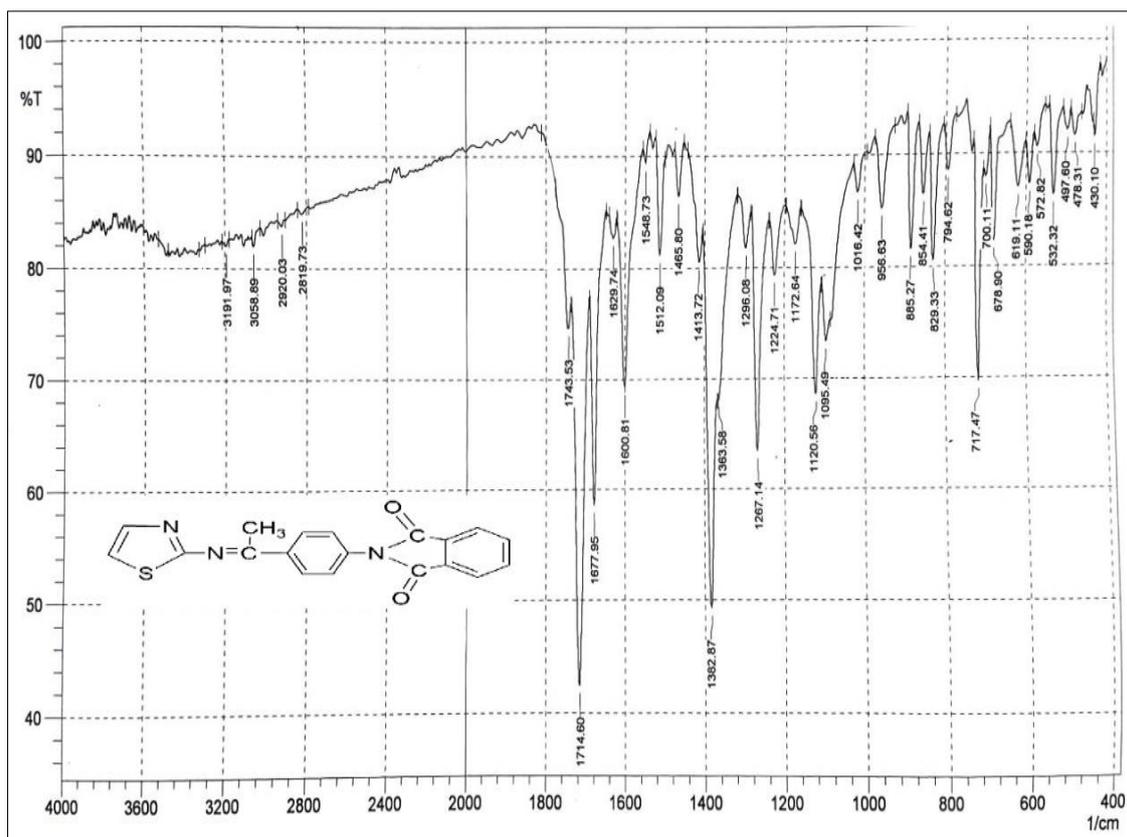
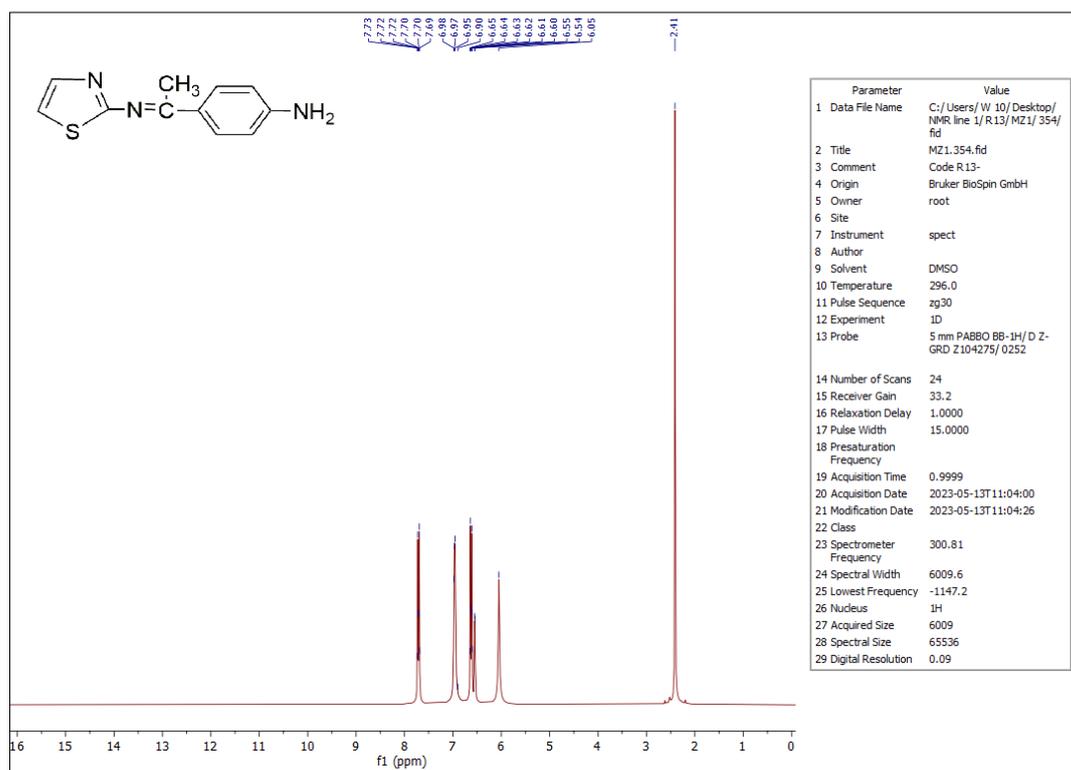
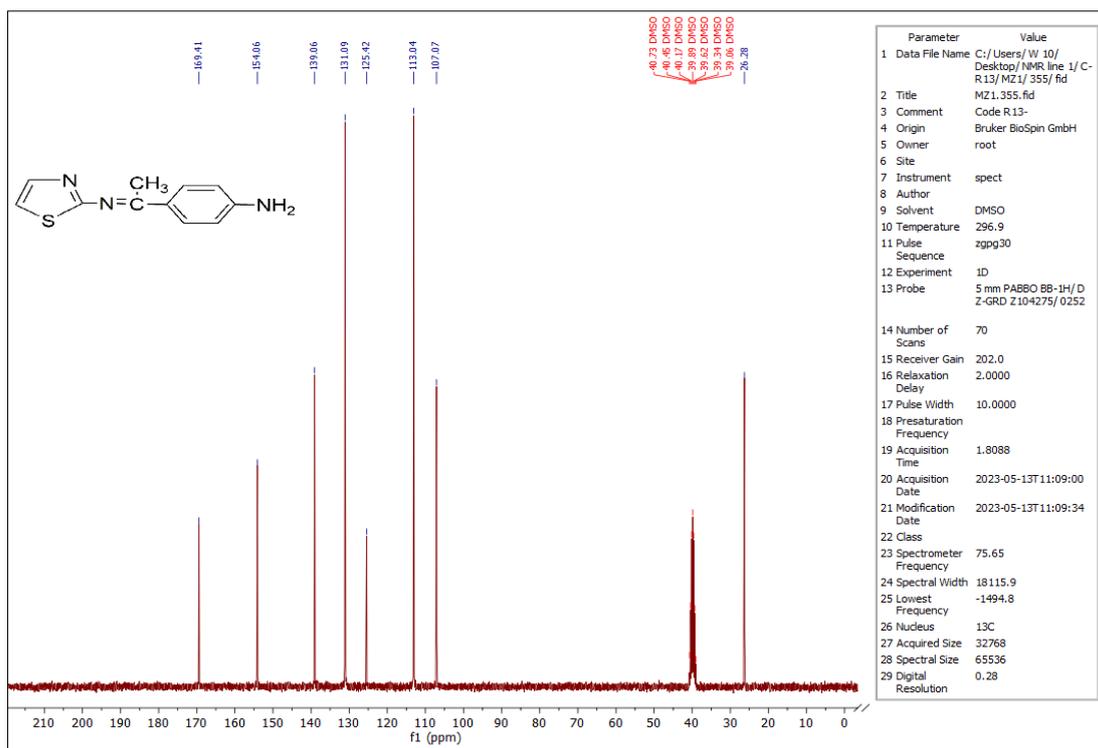
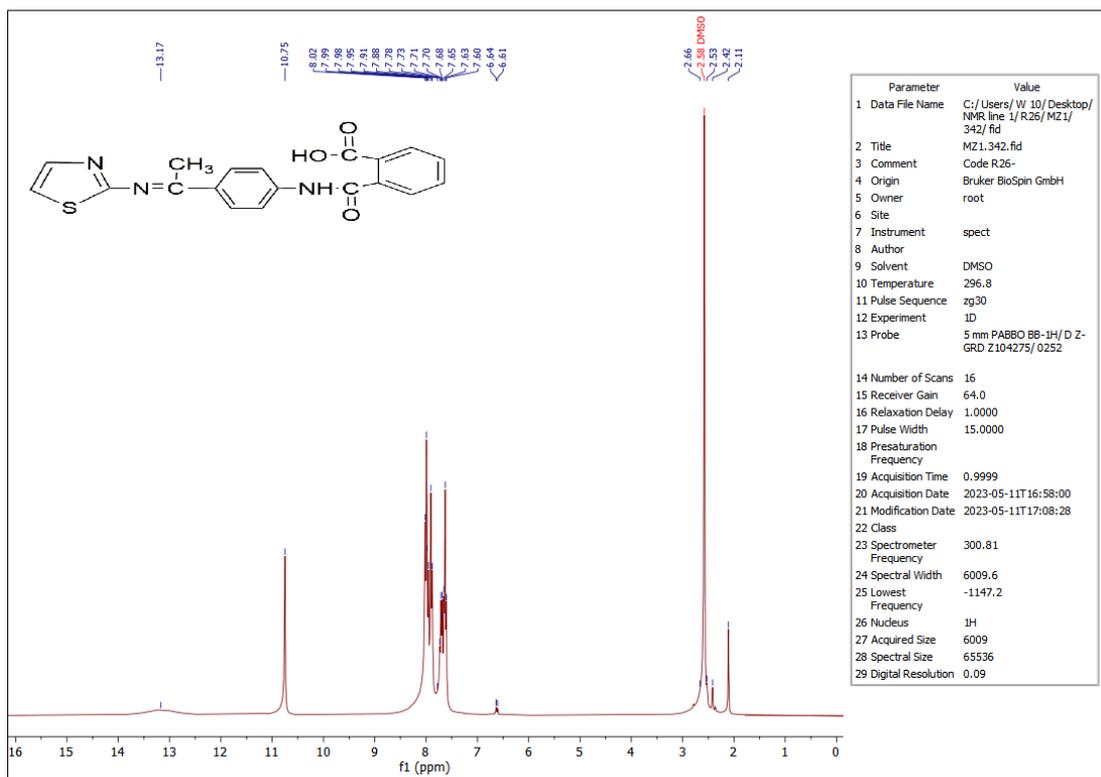
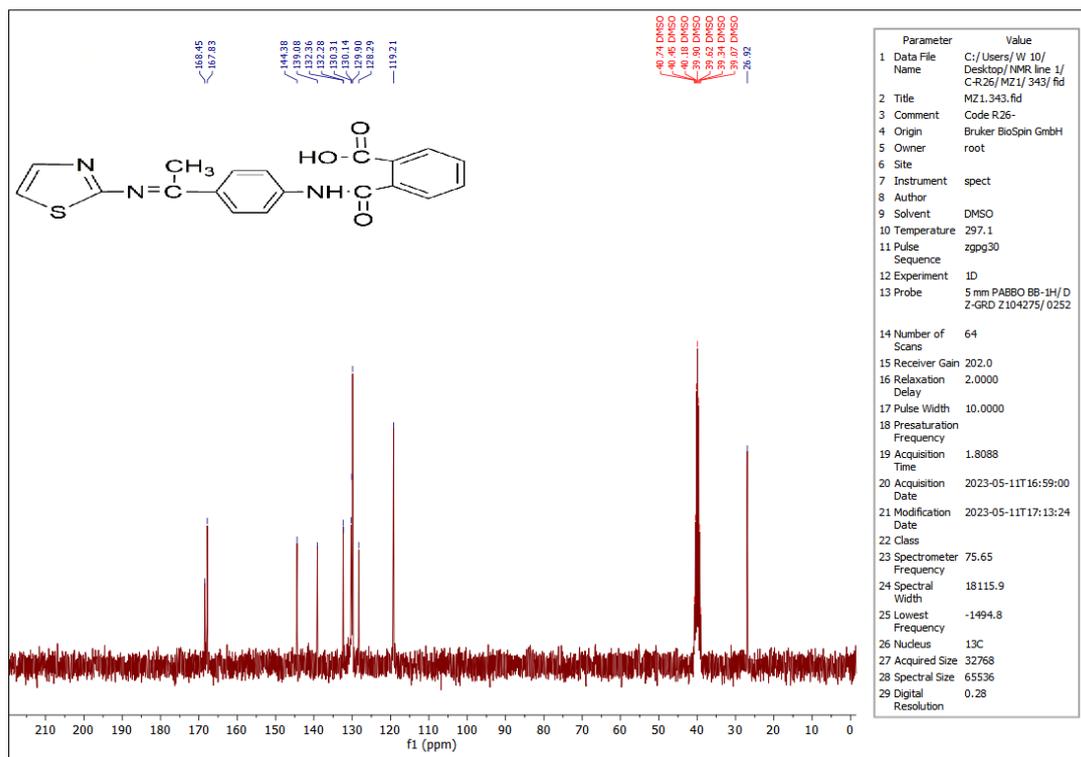
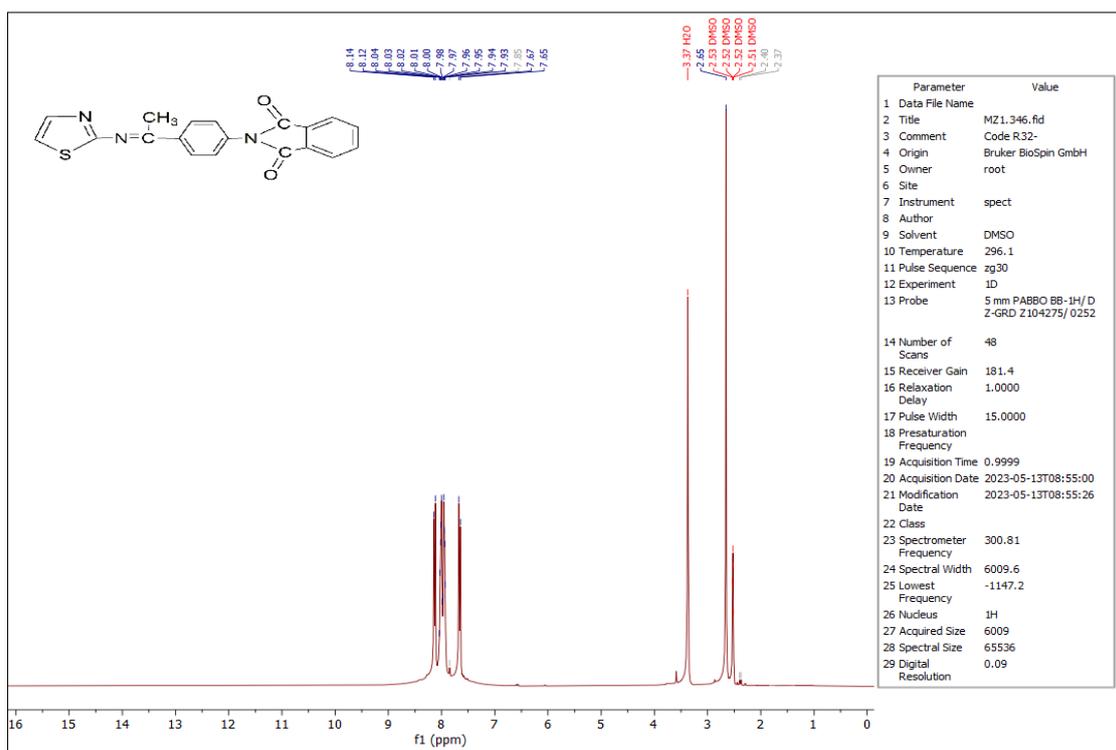
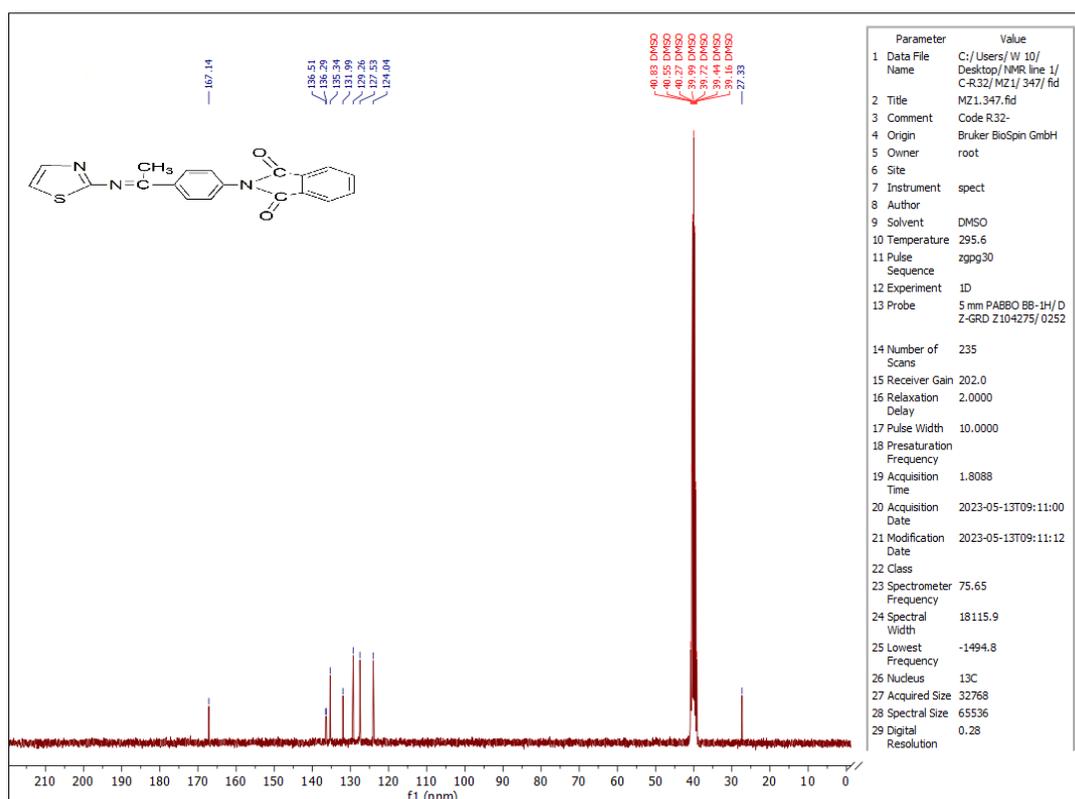


Figure 3: FT-IR spectrum of compound 10

Figure 4 : ¹H NMR spectrum data of compound 1Figure 5 : ¹³C NMR spectrum data of compound 1

Figure 6 : ¹H NMR spectrum data of compound 4Figure 7 : ¹³C NMR spectrum data of compound 4

Figure 8 : ¹H NMR spectrum data of compound 10Figure 9 : ¹³C NMR spectrum data of compound 10

4. Biological activity

Antibacterial activity for some of the prepared imides was evaluated against *Staphylococcus aureus* (gram-positive bacteria) and *E.coli* (gram-negative bacteria). We also

examined the antifungal activity of the produced imides against *Aspergillus niger* fungi, and the findings are shown in Table 9. Antipyriliminomaleimide **15** and thiazolyl iminotetrachlorophthalimide **16** were tested and found to have high antifungal and antibacterial activity against both types of bacteria. The imides, on the other hand, had moderate to good antibacterial and antifungal activity.

Table 9 : Antibacterial and antifungal activities of the tested prepared compounds (**11-18**) based on mm of the inhibition zone

No.	Inhibition zone		
	<i>Staphylococcus aureus</i>	<i>Escherichia coli</i>	<i>Aspergillus niger</i>
11	14	14	13
12	14	14	15
15	24	22	27
16	22	24	23
17	16	14	14
18	14	16	14
Amoxicilline	9	8	9
DMSO	0	0	0
7-10 = Weak, 11-15 = Moderate, 16-19 = Good, More than 20 = High			

5. Conclusion

The synthesis of nine new cyclic imides has been achieved successfully. The target products were isolated in yields ranging from 75 to 92%. These compounds contain three biologically active moieties (cyclic imide, heterocycle, and Schiff base). Our study practically proved the expected good antimicrobial activity of the synthesized compounds through the results of the antimicrobial activity. In general, the prepared imides possess good antibacterial and antifungal activities, especially compounds **15** and **16**, which showed promising results.

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