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Synthesis, characterization of amic acids and cyclic imides derived from acriflavine and evaluation of their antibacterial and antioxidant activity

Malath Khalaf Rasheed [a,*], Deena Saady Mohammed Subhi [b], Amenh Mohammed Abdulrahnan [c]

[a] Department of Chemistry, College of Education, University of Samarra, Samarra, Iraq
[b] Department of Chemistry, College of Education for Women, University of Tikrit, Tikrit, Iraq
[c] College of Medicine, Aliraqia University, Baghdad, Iraq

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ABSTRACT

Amic acids derivatives (**1**–**4**) have been synthesized by reaction of acriflavine with some cyclic anhydrides (succinic, maleic, phthalic and naphthalic) in absolute ethanol. Cyclic imides derivatives (**5**–**8**) were prepared by cyclization of amic acid derivatives using anhydrous acetic acid and anhydrase sodium acetate. The structures of the synthesized compounds have been identified by using the physical and spectral methods such as FT-IR, ^{1}H NMR and GC-Mass. The antibacterial activity of the prepared compounds was tested against (*staphylococs aureus* and *pseudomonas aeruginosa*) and all compounds show moderate to good antibacterial activity. However, the maximum inhibition zone was 15 mm against *staphylococs aureus* and 18 mm against *pseudomonas aeruginosa*. Furthermore, the antioxidant activity of these compounds have showed high activity and the maximum antioxidant was recorded to be 0.789 using the concentration 200 μg/cm^{3}.

Selection and peer-review under responsibility of the scientific committee of the International Conference on Advanced Materials Behavior and Characterization.

1. Introduction

Heterocyclic organics have interestingly used in the organic, nanomaterials and inorganic fields [1–15]. The organic molecule acriflavin plays an important role in medical, industrial and antiseptic applications [16,17]. Coal tar is the precursor for this compound and this compound has its familiar from its uses as antiseptic drug or sleeping sickness through the first world war due to his trypanocidal activity against trypanosomes [18]. Furthermore, in terms of uses against microbes that infect animals, acriflavine dilute solution have been used in the treatment and prevention the microbial infections in fish by adding to water [19]. For the synthesis of natural products, drugs, agrochemicals and different types of polymers, cyclic imides are favoured precursors and essential starting materials. Any of the notable natural products with the imide motif are salfredin C-1, lamprolobine, cladoniamide A, julocrotine, palasimide and migrastatin [20–22]. Moreover, imides and their cyclic moieties have been previously used as starting materials for the synthesis of many important and bioactive natural products [23]. However, different methods for the synthesis have been used such as the dehydrative condensation at high temperature for the anhydride/acids with different amines or through the cyclization of an amic acid with the help of acidic reagents in the presence of specific promoter. However, the later method suffers from low atom efficiency and production of by-products [24–27]. The cycle imides are the raw materials to synthesize the medicines and the agricultural chemicals [28]. It used in industrial as additives for the oils [29], and their derivatives used for prepared of azo compounds [30], as compounds for growth of plants such as wheat [31]. The derivatives of imides as perylene compounds are used in making the solar cells [32]. Herein, we prepared novel amic acids derivatives and we examined their biological activity against *staphylococs aureus* and *pseudomonas aeruginosa* in addition to their antioxidant activity.

2. Experimental part

2.1. Materials and instrumentations

All chemicals were supplied by Fuka, Merek and Aldrich chemicals Co. and used as received. Uncorrected melting points were determined by using Electro thermal melting Apparatus 9300. Thin

* Corresponding author.
E-mail address: almalath74@gmail.com (M.K. Rasheed).

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layer chromatography (TLC) was used for monitoring the reaction and to check the purity. The FT-IR spectra for the prepared organic compounds were FTIR- measured in the frequency range 400–4000 cm^{-1} using FT-IR-8300 Shimadzu as potassium bromide disks. ^1H NMR spectra for the newly prepared compounds were monitored using Bruker- 400 instrument with TMS as internal standard. The mass spectra of some compounds were registered by using the apparatus of kind GC.Mass QP-2013.

2.2. Methods

2.2.1. Preparation of amic acids derivatives (*1–4*)

All amic acids were prepared by mixing of 0.1 mol of acriflavine with 0.2 mol from each of cyclic anhydrides (succinic, maleic, phthalic and naphthalic) in 20 ml of absolute ethanol with stirring at room temperature. The precipitate was filtered off and washed in ether and recrystallized from ethanol. The purity of the compounds **1–4** was determined with TLC by using hexane: ethyl acetate mixture with a ratio of 2:8 [31,33]. Physical properties of amic acids compounds as shown in Table 1, Scheme 1.

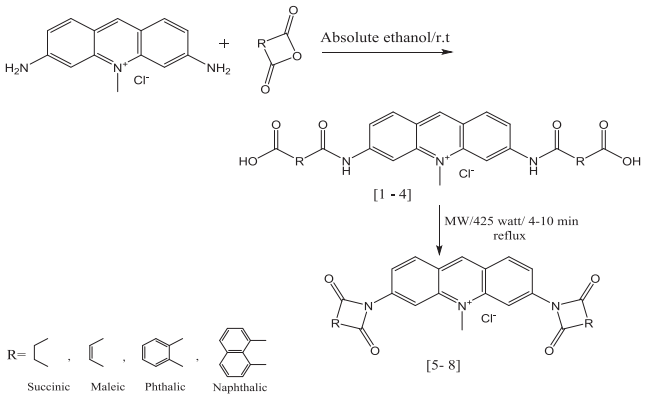
2.2.2. Preparation of cyclic imides derivatives (*5–8*)

Amic acids compounds (**1–4**) (0.01 mol) were dissolved in acetic anhydride 20 ml and 0.2 g anhydrase sodium acetate was added to the solution. The mixture was refluxed by using modified Microwave oven for 4–10 min (425 Watt). The resulted mixture was filtrated and recrystallized from ethanol to give the compounds (**5–8**). The purity of the compounds ***5–8*** was determined with TLC by using hexane: ethyl acetate mixture with a ratio of [2:8], respectively [34], see Rf in Table 1. Physical properties of cyclic imides compound as shown in Table 1.

3. Results and discussion

3.1. FTIr

The FT-IR spectra of compounds (**1–4**) showed disappearance of υ(NH_2) absorption that found in the started materials (acriflavine) and appearance of amidic υ(N—H) at the range 3320–3211 cm^{-1} in addition to υ(C═O) of acids and amide at the range 1768–1695 cm^{-1}. The spectra were also showed bonds at the range 3081–3055 cm^{-1} belonged to the υ(═C—H). Furthermore, the spectra were displayed an asymmetrical υ(C—N—C) at the range 1288–1012 cm^{-1}, and symmetrical υ(C—N—C) at the range 1030–1072 cm^{-1}. Additionally, the spectra showed the υ(C═C) and υ(C—N) appeared at 1558–1539 and 1427–1210 cm^{-1}, respectively [38–40]. The compounds (**5–8**) were showed of υ(C═O) imide at 1640–1668 cm^{-1} as showed in the Table 2. The Figs. 1–4 showed the FT-IR spectra for the compounds (**1**,**2**,**5**,**8**) [35,36].



Scheme 1. Synthesis steps of prepared compounds.

3.2. ^1H NMR spectra of compounds (*1*,*6*,*8*)

^1H NMR of the compound (**1**) in the DMSO d_6 solvent the was shown in Fig. 5. The spectrum shows a singlet signal at 10.90 ppm that assigned to the proton of O—H in acid and another singlet signal at 10.35 ppm that belonged to NH. The signals at in the range of 7.06–8.88 ppm are belonged to the protons of aromatic rings. Moreover, the spectrum shows the aliphatic protons as a singlet signal at 4.55 ppm belonged to CH_3 and at 3.30–3.47 ppm belonged to the protons of CH_2 group [37,38].

The ^1H NMR spectrum of compound (**6**) Fig. 6 showed many signals, at the range 7.17–8.88 ppm could be attributed to the protons of benzene ring. The signal at 6.37–6.54 ppm due to the protons of CH olefin in the five membered ring. The signal at 5.38 ppm could be assigned to protons of CH_3 while the signals at 2.5 and 3.3 ppm assigned to protons of DMSO and the water.

The ^1H NMR spectrum of compound (**8**) Fig. 7 showed multiplet signals at 6.93–8.05 ppm which could be attributed to the protons of benzene ring. The signal at 4.10 ppm due to the aliphatic protons of CH_3 [24,25].

3.3. The Mass spectra of compounds (*3*,*5*)

Figs. 8 and 9 exhibited the mass spectra of compounds (**3**,**5**) which showed the molecular weight of 555.5 g/mole for compound (**3**) and 423.5 g/mole for compound **5** which belongs to their molecular ions [41,42].

3.4. Antibacterial activity

Antibacterial activity of synthesized compounds was evaluated in vitro by using the diffusion method [43] against two microorganisms; *staphylococcus aurous* (G+) and *pseudomonas aeruginosa* (G−). The compounds were tested at a concentration of 5, 10 and

Table 1
Physical properties and Rf determined from TLC of **1–8.**

Comp.No.	CompoundsFormula	Molecular Weight(g/mole)	Color	M.P °C	Yield %	Rf
1	$C_{22}H_{22}N_3O_6Cl$	459.5	Yellow	125–127	**79**	**0.71**
2	$C_{22}H_{18}N_3O_6Cl$	455.5	Orange	138–140	**75**	**0.67**
3	$C_{30}H_{22}N_3O_6Cl$	555.5	Yellow	130–131	**85**	**0.63**
4	$C_{38}H_{26}N_3O_6Cl$	655.5	White	262–264	**82**	**0.77**
5	$C_{22}H_{18}\ N_3O_4Cl$	423.5	Brown	194–196	**70**	**0.84**
6	$C_{22}H_{14}N_3O_4Cl$	419.5	Dark Brown	202–204	**65**	**0.65**
7	$C_{30}H_{18}N_3O_4Cl$	519.5	Brown	255–257	**76**	**0.72**
8	$C_{38}H_{22}N_3O_4Cl$	619.5	Brown	108–110	**61**	**0.88**

Table 2
FT-IR Spectral Data for the prepared compounds (**1**–**8**).

No. of Comp.	ν(N—H) amide	ν(C—H) arom	$\nu_{asy.}(CH_2)$ $\nu_{sy.}(CH_2)$	ν(C=O) imide amide	ν(C=C) arom	ν(O—H)	ν(C—N—C) $\nu_{asy.}$,$\nu_{sy..}$
1	3176	3012	2927	——	1587	3315	1288
			2844	1647			1012
2	3178	3013	2937	——	1533	3313	1180
			2831	1641			1130
3	3213	3012	2977	——	1587	3320	1230
			2844	1650			1030
4	3212	3015	2937	——	1581	3340	1260
			2887	1647			1035
5	——	3089	2933	1685	1583	——	1205
			2844	——			1170
6	——	3058	2997	1663	1542	——	1215
			2820	——			1012
7	——	3056	2994	1659	1543	——	1240
			2887	———			1040
8	——	3068	2978	1660	1540	——	1270
			2888	———			1045

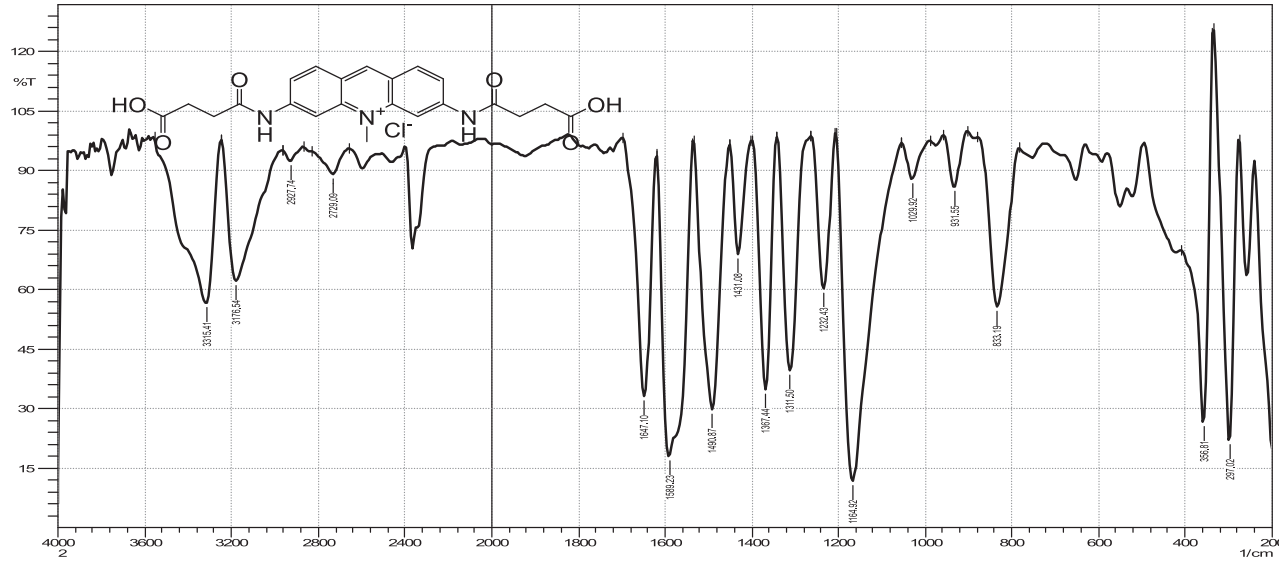


Fig. 1. FT-IR Spectrum of compound (**1**).

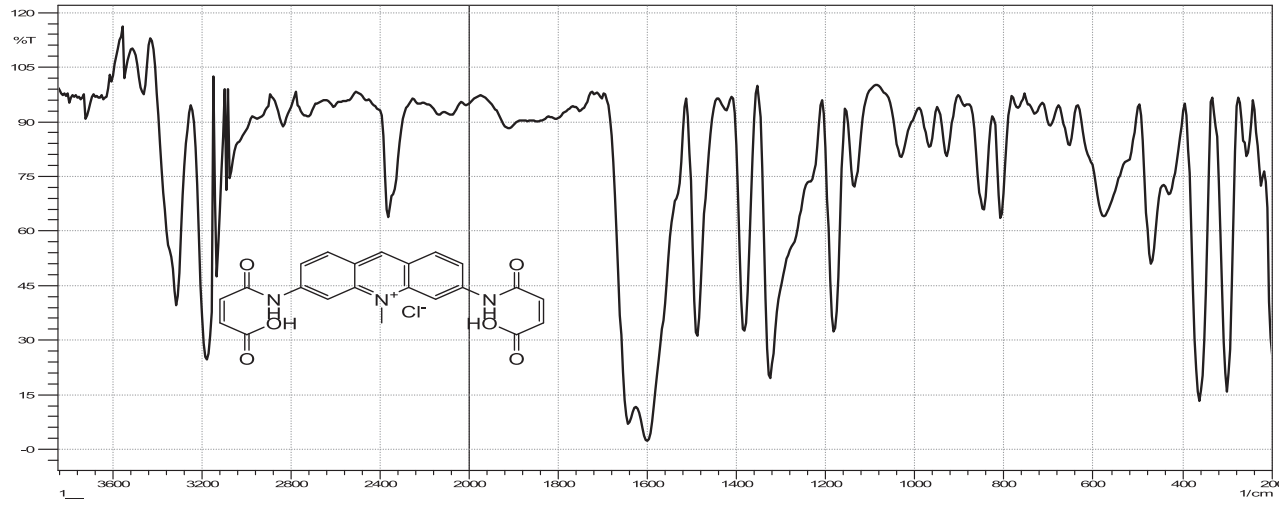


Fig. 2. FT-IR Spectrum of compound (2).

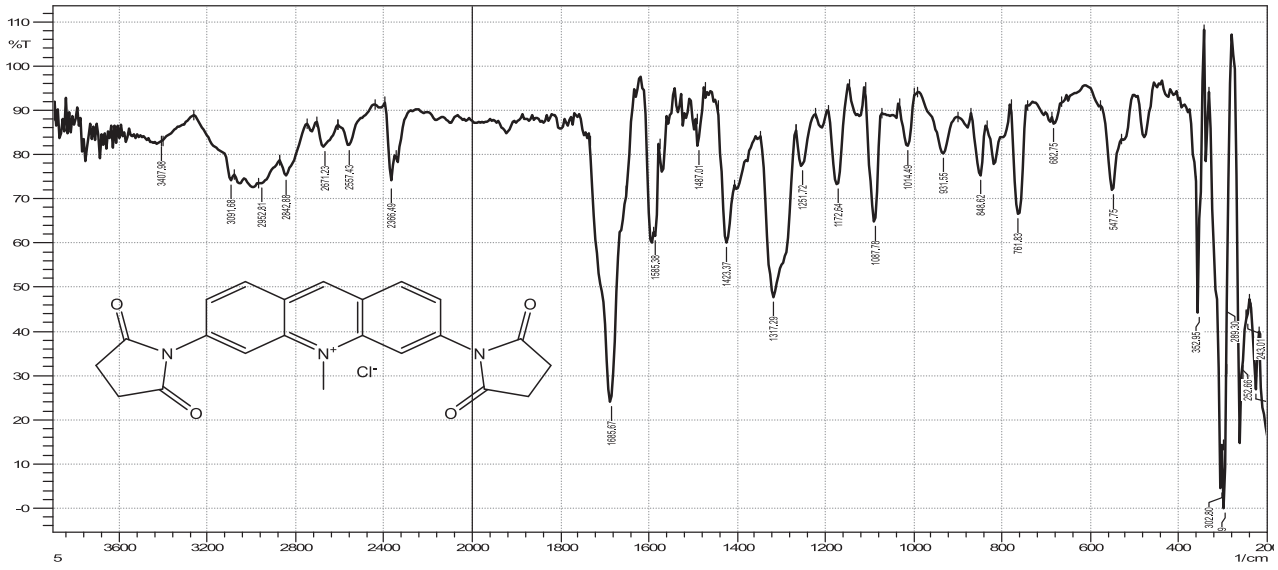


Fig. 3. FT-IR Spectrum of compound (5).

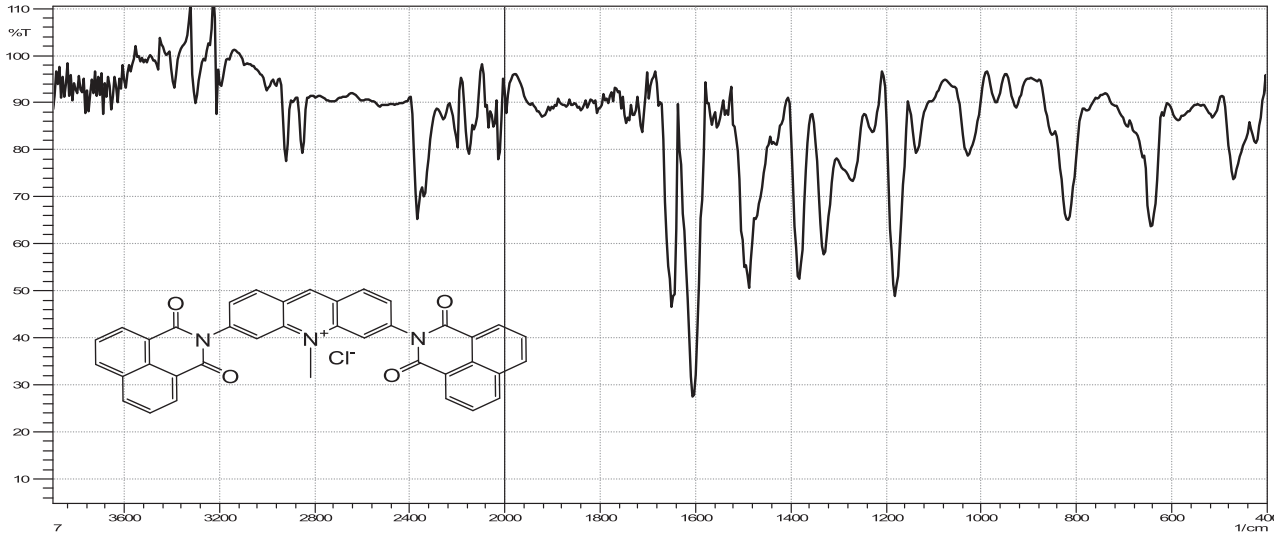


Fig. 4. FT-IR Spectrum of compound (8).

15 mg/ml in dimethylsulfoxide. The results revealed that compounds showed inhibition activities compared with the standard drug (St.) neomycin sulfate 15 mg/ml, as shown in Table 3.

3.5. Antioxidant activity

The total antioxidant susceptibility of synthesized compounds was estimated as antioxidants in vitro. The results showed that the prepared compounds were good compared to the standard (St.) Vit.C in concentrations 25, 50, 100, 150 and 200 μg/cm^3, as shown in Table 4 [44].

4. Conclusions

This study is aims to synthesize, characterize and the antibacterial and antioxidant activities of new amic acid and cyclic imides derivatives with the hope of discovering new structures serving as potential broad-spectrum antibacterial agents and antioxidant agents. The structural analysis was done by spectroscopic methods. Amic acid, cyclic imides and its derivatives play very important role in medicinal and pharmaceutical applications. Some of the tested products showed moderate activity as the results were reported.

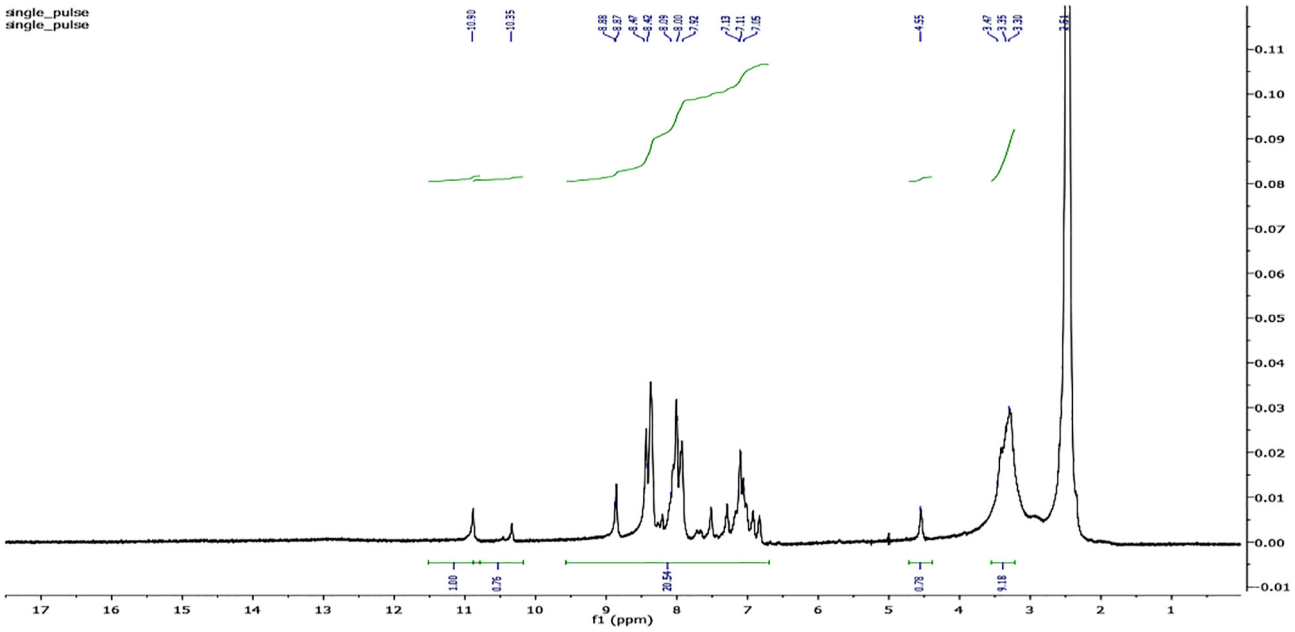


Fig. 5. ^{1}H NMR Spectrum of compound (1).

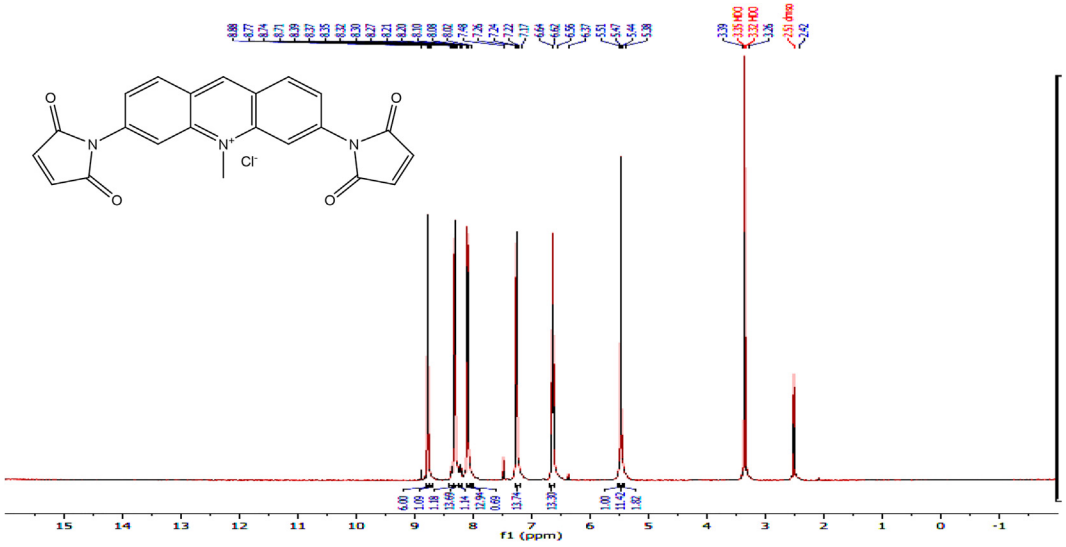


Fig. 6. ^{1}H NMR Spectrum of compound (**6**).

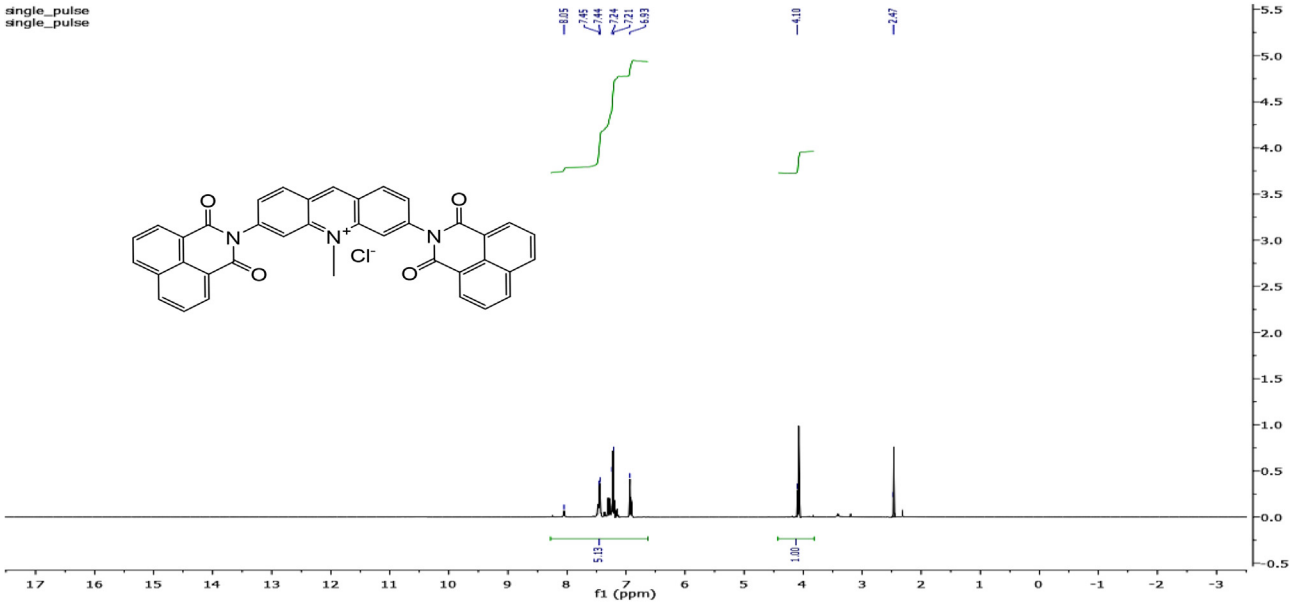


Fig. 7. ^{1}H NMR spectrum of compound (**8**).

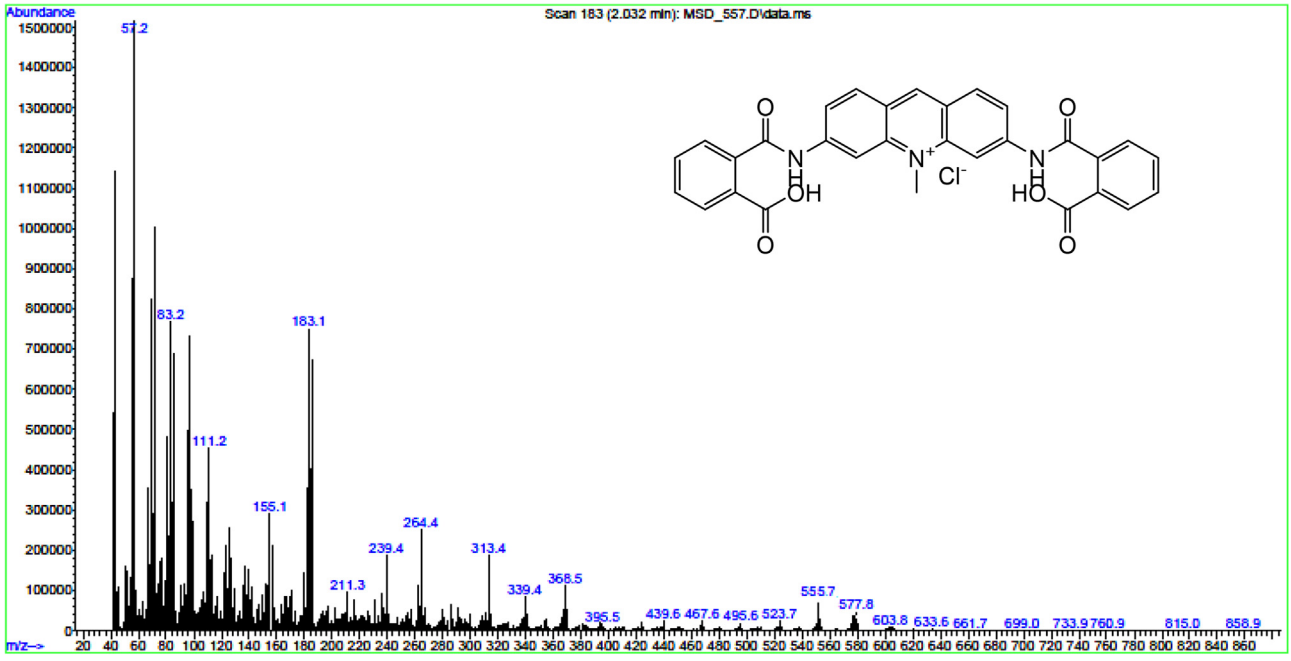


Fig. 8. Mass Spectrum of compound (**3**).

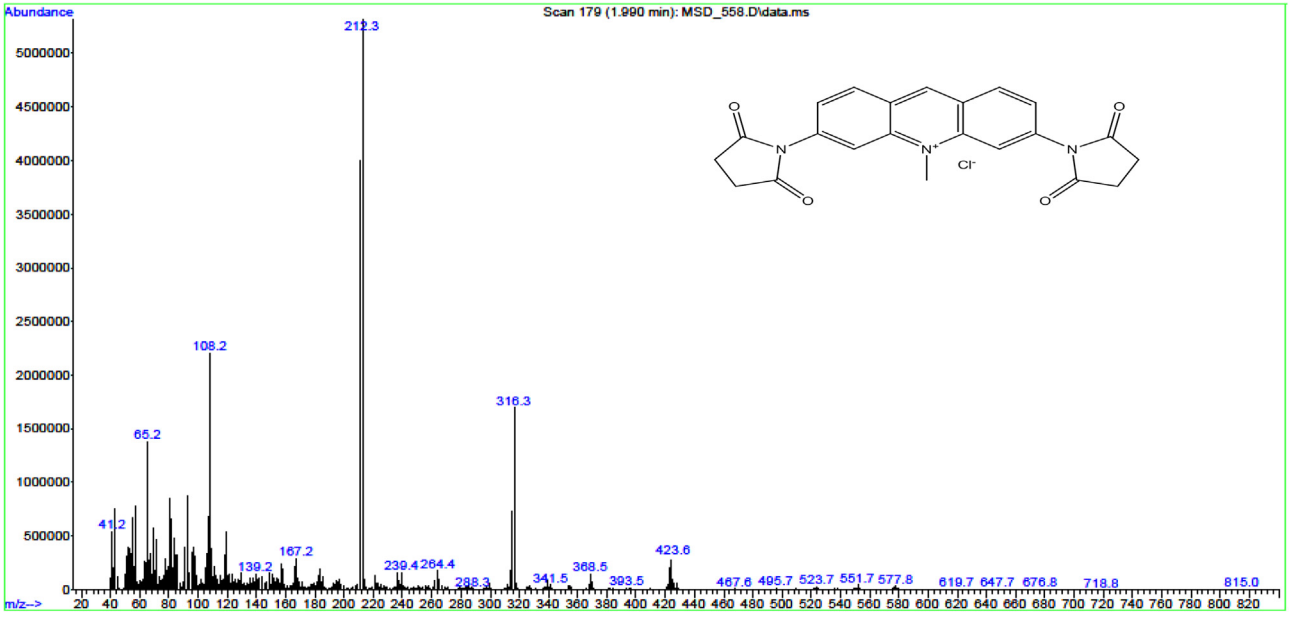


Fig. 9. Mass spectrum of compound (**5**).

Table 3
Antibacterial activity of the prepared compounds.

Zone of inhibition (mm)									
Staphylococcus aurous (G +)					*pseudomonas aeruginosa* (G−)				
Comp.	a	b	c	St.	Comp.	a	b	c	St.
1	—	11	15	20	1	10	12	15	25
2	—	10	15	20	2	9	11.5	13	25.2
3	11	15	18	21.3	3	9.5	10.7	12.2	25
4	—	—	9	20.1	4	8	9	12	25
5	8	10	14	20.1	5	11	9	13.5	25
6	5	12	18	21	6	9	10.2	12	25
7	8	12	15	20.1	7	8	10.2	13	25.1
8	10	15	18	20.5	8	10	10	15	25

a = 5 mg/ml, b = 10 mg/ml, c = 15 mg/ml. St. = Neomycin sulfate 15 mg/ml
Highly active = inhibition zone > 20 mm. Moderately active = inhibition zone 11–20 mm Slightly active = inhibition zone 5–10 mm. No inhibition (—).

Table 4
Antioxidant data of synthesized compounds and the standard (St.) Vit.C.

Comp.No.	25 μg/cm^3	50 μg/cm^3	100 μg/cm^3	150 μg/cm^3	200 μg/cm^3
1	0.202	0.308	0.321	0.322	0.355
2	0.322	0.354	0.359	0.411	0.455
3	0.241	0.326	0.463	0.582	0.701
4	0.222	0.363	0.458	0.555	0.355
5	0.201	0.224	0.436	0.516	0.245
6	0.132	0.325	0.225	0.587	0.479
7	0.245	0.221	0.352	0.215	0.775
8	0.201	0.311	0.353	0.445	0.789
St.Vit.C	0.225	0.363	0.579	0.737	0.965

CRediT authorship contribution statement

Malath Khalaf Rasheed: Conceptualization, Investigation, Validation. **Deena Saady Mohammed Subhi:** Visualization, Formal analysis. **Amenh Mohammed Abdulrahnan:** Data curation, Methodology, Investigation.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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