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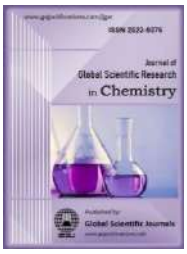

Synthesis, Study the Antibacterial Activity and Liquid Crystal Phase of Some New 2,3-Dihydroquinazoline-4-one Compounds Derived From Triethyl Benzene-1,3,5-Tricarboxylate as a Basic Nucleus

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ABSTRACT

This study included the preparation of hydrazone derivatives (S2-S6) by reacting hydrazide compound (S1) with aromatic aldehyde substitutes and then reacting them with anthranilic acid to obtain a six-ring derived from 2,3-dihydroquinazoline-4-one (S7-S11). The validity of the prepared compounds was confirmed using physical techniques (color, melting point, radio frequency) and spectroscopic methods. (FT-IR infrared spectrum, carbon-proton NMR spectrum, 13C-NMR, and 1H-NMR). The biological activity of some of these compounds and two types of Gram-positive and Gram-negative bacteria (Staphylococcus aureus and Escherichia coli) was also studied. The properties of liquid crystals were also investigated. And the changes that occur on some compounds using hot polarized optical microscopy.

1. Introduction

Due to their widespread occurrence in nature and their role in synthesizing several organic molecules vital to life's fundamental components, heterocyclic rings are very significant. Additionally, they can be found in various forms in sugars and their derivatives. In addition to carbon atoms, heterocyclic molecules also contain nitrogen, oxygen, and sulfur atoms in their structure [1]. When a carbonyl group is present at position 4 in hydro quinazoline-4-one, a ring containing two nitrogen atoms and a carbonyl group, a six-ring known as 2,3-dihydroquinazoline-4-one, is created [3].

2,3Dihydroquinazoline-4-one compounds are of great biological and pharmacological interest because they are closely related to medicinal chemistry and prepare many drugs and antimicrobials (4). What has recently drawn attention is the great interest of researchers in the importance and bioavailability of these compounds, as some of them, in addition to their antispasmodic, antispasmodic, and anti-gastrointestinal effects, also show analgesic, antibacterial, antifungal (5), and anti-enteric activity. Tuberculosis medications and depression treatments also showed good anti-tumor

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activity (6). Hydrazone contains the azomethine group (-HC=N-) and is usually characterized by its yellow color. They are named after the chemist Hugo Schiff (7). They are imines containing a nitrogen atom attached to an aryl group. Or an alkyl group and not a hydrogen atom, so the connection of the organic part to the nitrogen atom has the effect of increasing the stability of the resulting imine and preventing its decomposition or polymerization, and it has the general formula (R1R2C=N-R3) (8). Schiff bases are used as antidotes for many diseases, including antioxidant and antibacterial ones because the compounds contain an azomethine group (HC=N), which has a wide range of biological activities (9). In addition, it is effective against cancer (10). Liquid crystals Materials with a liquid look are called liquid crystals. However, their molecular arrangement is similar to that of crystals (11). It is a condition in the middle between a stable liquid and a crystalline solid. According to specific theories, liquid crystals represent the fourth state of matter (12). Liquid crystals are a transitional condition between the isotropic or liquid phase, where molecules are free to move, and the solid phase, which displays perfect molecular order in position and orientation (13).

2. Materials and methods

2.1. Chemicals used

Chemicals prepared from Aldrich, BDH, Thomas, Fluka, and Merck were used.

2.3 Devices used

Melting points were measured with a thermoelectric melting point apparatus. KBr disks at 400-4000 cm^{-1} scale, Shimadzu FT-IR 8400S spectrophotometer, Bruker equipment running at 400 MHz for ^{1}H-NMR and ^{13}C-NMR spectra. Fluka silica gel plates, with a thickness of 0.2 mm, were used in thin-layer chromatography (TLC). UV light achieved visibility after fluorescent silica gel G activated the plates.

2.3. Preparation of Hydrazone derivatives (S_2-S_6).

Mix (0.003 mol, 0.8 g) of 1,3,5-tricarbonyl hydrazide benzene in a minimal amount of ethanol with the benzaldehyde substituted (0.009 mol), then add three drops of glacial acetic acid and reflux the mixture as (12-15) hour, the solution is cooled, then concentrated, the solution is filtered, the residue is dried, and it is recrystallized from ethanol (14). The physical properties of prepared compounds(S_2-S_6) are shown in Table (1).

2.3. Preparation of 2,3-dihydroquinazoline-4-one derivatives (S_7-S_{11}).

Dissolve (0.004 mol) of the prepared hydrazones (S_2-S_6) in (10 ml) of 1,4-dioxane with (0.012 mol,1.7 g) of 2-aminobenzoic acid dissolved in (10 ml) of the same solvent (15). It was left to reflux for (12-15) hours, and the filtrate was cooled to room temperature and then washed using a sodium bicarbonate solution (10%). It was washed with water, the residue was dried, and it was recrystallized with the solvent 1,4-dioxane. Table (1) shows the physical properties of the compounds (S_7-S_{11}).

2.4. Biological activity study

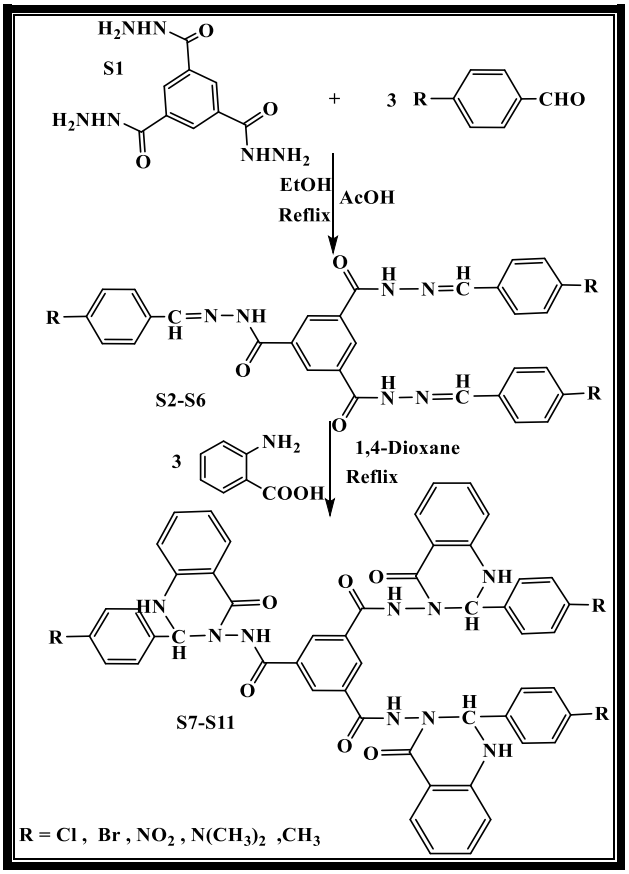
Two colonies of pure bacterial isolates of both Gram-positive and Gram-negative bacteria were transferred from the solid culture medium to test tubes containing (5 ml) distilled water using heat-sterilized carriers. The tubes were incubated at 30°C. Temperature (37 degrees Celsius) for 16-20 hours and then diluted with physiological solution until the turbidity reaches the standard turbidity level to obtain a cell count of approximately (1.5 x 108) cells/ml. Chemical solutions of some of the prepared compounds were prepared using the solvent dimethyl sulfoxide (DMSO)[16,17]. The concentration of each substance was (0.01, 0.001, 0.0001) mg/ml, and each solid derivative (0.1 g) was dissolved in (10 ml) medium. Of (DMSO) to obtain a concentration of (0.01 mg/ml), then remove (1 ml) of the solution with a concentration of (0.01 mg/ml) and add (9 ml) of (DMSO) solvent. Add to obtain a solution at a concentration of (0.001 mg/ml), then withdraw (1 ml) and (9 ml) DMSO from the solution at a concentration of (0.001 mg/ml) (18,19). Add the solvent to obtain a solution of (0.0001 mg/mL).

2.5. Study of the liquid crystalline properties of some prepared compounds:[20]

The structures of some of the prepared compounds were examined using a polarizing microscope equipped with an E-PLAN 10X/0.25 160/0.17 lens, an electronic thermometer to detect temperature, and a MEIJI hot-stage heater (made in the USA). After improvements, it now has a 38MP FHD V6 camera with a 20x zoom. All compounds under investigation were produced using thin-film technology, samples were extensively inspected using magnifying glasses and automated thermal imaging cameras, and textural aspects of the compounds were photographed.

3. Results and discussions

This study involved the preparation of several compounds, as the scheme (1) illustrates.



Scheme (1): Path of the Ready Compounds (S2-S11)

3.1 Characterization of 2,3-Dihydrazone-4-one derivatives (S2-S6)

When studying the infrared spectrum (FTIR) of the prepared hydrazone compounds [S2-S6], it was noted that the two stretching bands of the amine group (NH_2) and C=O of aldehyde belonging to the hydrazide compound had disappeared. The stretching bands appearing in the range (1595-1612) cm^{-1} belong to isomethene (C=N) groups, unique to peel compounds and evidence of product formation. In addition to these bands, an absorption band in the range of (3075-3031) cm^{-1} also appears, and it belongs to the stretching of aromatic bonds (C-H). The lines appear due to the expansion of olefin bonds (=CH) in the range (3126-3176) cm^{-1}. Two absorption bands belonging to aromatic bonds (C=C) appear in the range (1517-1569) cm^{-1} and (1454-1493) cm^{-1}. The lines appear due to the stretching of the amide bond (C=O) in the range (1666-1672) cm^{-1}. In addition to the appearance of an absorption band in the range (3275-3231) cm^{-1} due to the

expansion of the (N-H) bonds (21), as shown in Table (1) and Figure (1,2).

When studying the ^{1}H-NMR spectrum of compound (S2), a single signal occurs at this location (6.84 ppm), which is caused by the protonation of the azomethine group (N=CH). One signal also appears at (11.28) ppm belonging to the amino group (N-H). The multiple signals ranged between (7.38-8.77) ppm due to the protons of the benzene ring. As for the solvent DMSO-d^6, a signal appeared at (2.51) ppm. As in Figure (3).

3.2 Characterization of 2,3-dihydroquinazoline-4- one derivatives (S_7-S_{11})

When studying the infrared spectrum (FTIR) of the 2,3-dihydroquinazoline-4-one compounds (S7-S11), it was observed that the two stretching bands disappeared from one to the something groups (C=N), and the appearance of two double stretching bands in the range (2941-2977) cm^{-1}and (2887-2927) cm^{-1} that belong to the squeezing stretch (C-H) hydroquinazoline ring. In addition to these bands, an absorption band in the range of (3034-3078) cm^{-1} also appears, and it belongs to the stretching of aromatic bonds (C-H). Two absorption bands belonging to aromatic bonds (C=C) appear in the range (1517-1569) cm^{-1} and (1454-1493) cm^{-1}. The lines appear due to the carbonyl bond (C=O) expansion in the range (1654-1639) cm^{-1}. In addition to the appearance of an absorption band in the range (3211-3321) cm^{-1} due to the expansion of (N-H) bonds **(22)**, as shown in Table (1) and Figure (4,5).

When studying the ^{1}H-NMR spectrum of compound (S8), a single spark occurs at this location (5.96) ppm, caused by the protonation of the (CH) group of the quinazoline ring. One signal also appears at (6.82) ppm, belonging to the ring's amino group (NH) the same. One signal also appears at (11.67) ppm belonging to the amino group (NH) amide. The multiple signals range between (7.24-8.37) ppm due to the protons of the benzene ring. As for the solvent DMSO-d^6, a signal appeared at (2.51) ppm. As in Figure (6).

When studying the ^{1}H-NMR spectrum of compound (S9), a single spark occurs at this location (5.76) ppm, caused by the protonation of the (CH) group of the quinazoline ring. One signal also appears at (6.14) ppm, belonging to the ring's amino group (NH) the same. One signal also appears at (10.18) ppm belonging to the amino group (NH) amide. The multiple signals range between (6.82-8.54) ppm due to the protons of the benzene ring. As for the solvent DMSO-d^6, a signal appeared at (2.51) ppm. As in Figure (7).

3.3 Evaluation of biological activity of prepared compounds

Staphylococcus aureus and ***Escherichia coli*** are used in this study to examine the biological activity of the chemicals generated. Heterocyclic compounds have distinct biological effects that target both Gram-positive and Gram-negative bacteria. These bacteria were selected due to their reputation for causing various illnesses. Furthermore, these microbes exhibit different patterns of antibiotic resistance **(23).** The bioactivity of the resultant compounds was assessed by measuring the inhibitory zone width and applying the agar well diffusion technique **(24).** The outcomes demonstrated that the synthesized compounds may, to differing degrees, limit the development of both Gram-positive and Gram-negative bacteria. This compound has remarkable inhibitory action against ***Staphylococcus aureus*** and substantial inhibitory activity against ***Escherichia coli*** **(25,26).**

3.4 Discussion and identification of some liquid crystal phases

The values of the thermal transition degrees of most of the prepared compounds' liquid and isotropic crystalline phases were assigned. The nature of these transitions was studied, and liquid crystal forms were diagnosed using a polarized light microscope with a heater, as shown in Table (4). Compound [S2] showed two transitions when studied with a polarized light microscope; the first transition is due to the melting point, and the second is due to the transition from the crystalline phase to the Smectic phase (S). As shown by the histological structure in Figure (10). As for the compound (S3), which showed three transitions, the first transition is due to the melting point, the second transition is due to the transition from the crystalline phase to the Smectic phase (S), and the last transition is the transition from the Smectic phase (S) to the Nematic phase. The histological structure is shown in Figure **(11,12).** As for the compound (S9), it showed three transitions: the

first transition is due to the melting point, the second transition is due to the transition from the crystalline phase to the Smctic phase (S), and the last transition is the transition from the Smectic phase (S) to the Nematic phase (N). The histological structure is shown in Figure (13,14).

Table 1: Some physical properties of some prepared compounds (S_2-S_{11})

Comp No	R	Molecular Formula	m.p. °C	Yield %	R.T hour	R_f	Color
S_2*	4-Cl	$C_{30}H_{21}Cl_3N_6O_3$ 619.89	219-230	77	15	0.88	Light yellow
S_3*	4-Br	$C_{30}H_{21}Br_3N_6O_3$ 753.25	298-310	63	12	0.62	Yellow
S_4	4-NO_2	$C_{30}H_{21}N_9O_9$ 651.55	314-316	74	15	0.85	Dark yellow
S_5*	4-$N(CH_3)_2$	$C_{36}H_{39}N_9O_3$ 645.77	280-305	88	15	0.73	Orange
S_6	4-CH_3	$C_{33}H_{30}N_6O_3$ 558.64	312-3114	75	15	0.87	Yellow
S_7	4-Cl	$C_{51}H_{36}Cl_3N_9O_6$ 977.26	319-317	84	15	0.95	Dark yellow
S_8*	4-Br	$C_{51}H_{36}Br_3N_9O_6$ 1110.62	250-280	84	14	0.79	Yellow
S_9*	4-NO_2	$C_{51}H_{36}N_{12}O_{12}$ 1008.92	220-262	80	14	0.88	Dark brown
S_{10}	4-$N(CH_3)_2$	$C_{57}H_{54}N_{12}O_6$ 1003.14	285-287	66	12	0.64	Brown
S_{11}	4-CH_3	$C_{54}H_{45}N_9O_6$ 916.01	310-312	87	12	0.96	Dark brown

NOTE: compounds marked with a star have had their properties studied its liquid crystalline.

Table 2: Ft-IR absorption results for prepared compounds (S_2-S_{11})

Comp. No.	R	ν(N-H)	ν(C=O)	ν(C-H) Arom.	ν(C-H) Olph.	ν(C=N)	ν(C=C) Arom.	Others
S_2	4-Cl	3272	1668	3060	3134	1595	1517,1477	ν (C-Cl) 740
S_3	4-Br	3275	1670	3058	3184	1598	1500,1448	ν (C-Br) 524
S_4	4-NO_2	3231	1667	3075	3147	1607	1535,1469	ν(NO_2).*asy*.(1516) *sym*.(1325)
S_5	4-$N(CH_3)_2$	3267	1672	3031	3126	1603	1569,1481	ν (C-N) 1237
S_6	4-CH_3	3256	1666	3067	3156	1612	1543,1451	--
Comp. No.	R	ν(N-H)	ν(C-N)	ν(C-H) Arom.	ν(C-H) Aliph.	ν(C=O)	ν(C=C) Arom.	Others
S_7	4-Cl	3291	1234	3034	2964,2912	1642	1548,1472	ν (C-Cl) 767
S_8	4-Br	3311	1251	3078	2972,2902	1639	1569,1508	ν (C-Br) 598
S_9	4-NO_2	3321	1246	3051	2941,2887	1651	1552,1457	ν(NO_2).*asy*.(1521) *sym*.(1336)
S_{10}	4-$N(CH_3)_2$	3287	1238	3073	2977,2921	1649	1561,1473	--
S_{11}	4-CH_3	3211	1222	3037	2968,2900	1654	1548,1456	--

Table 3: Biological efficacy of produced substances and control methods (Measured in millimeters of inhibition.

Comp. No.	*E. Coil Conc. mg/ml*			*Staph. Aureus Conc. mg/ml*		
	0.01	**0.001**	**0.0001**	**0.01**	**0.001**	**0.0001**
S6	15	10	5	30	20	15
S7	21	15	11	25	15	10
S9	20	15	10	17	10	5
S10	18	14	5	16	12	7
Amoxicillin	23	18	15	21	18	15

Table 4: Liquid crystal phase transitions in a device Mic.sc for prepared vehicles.

Apparatus	NO.	Crystal	Smectic A	Nematic	ΔT_{SA}	ΔT_N
Microscope	S_2	219	230	------	11	------
	S_3	298	310	316	12	18
	S_5	280	305	315	25	35
	S_5	224	280	300	56	76

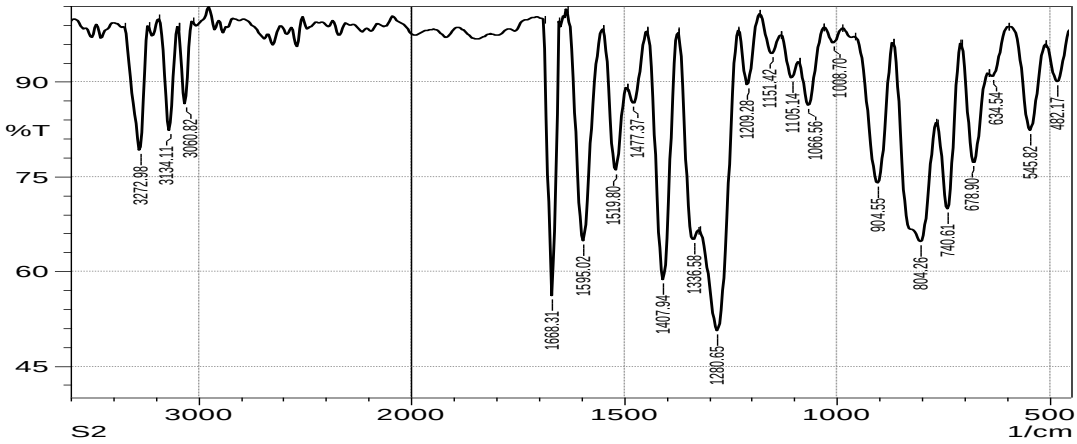


Figure 1: FT-IR spectra of coumpond (S_2).

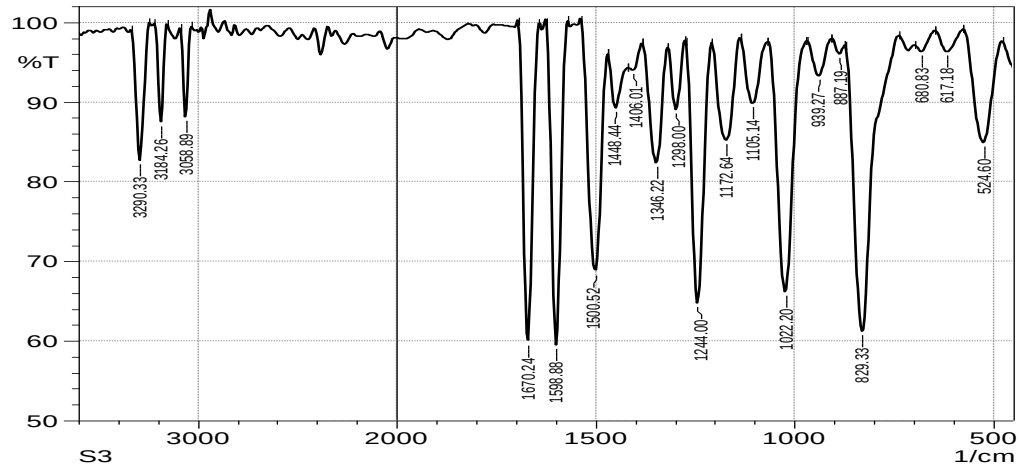


Figure 2: FT-IR spectra of compound (S3).

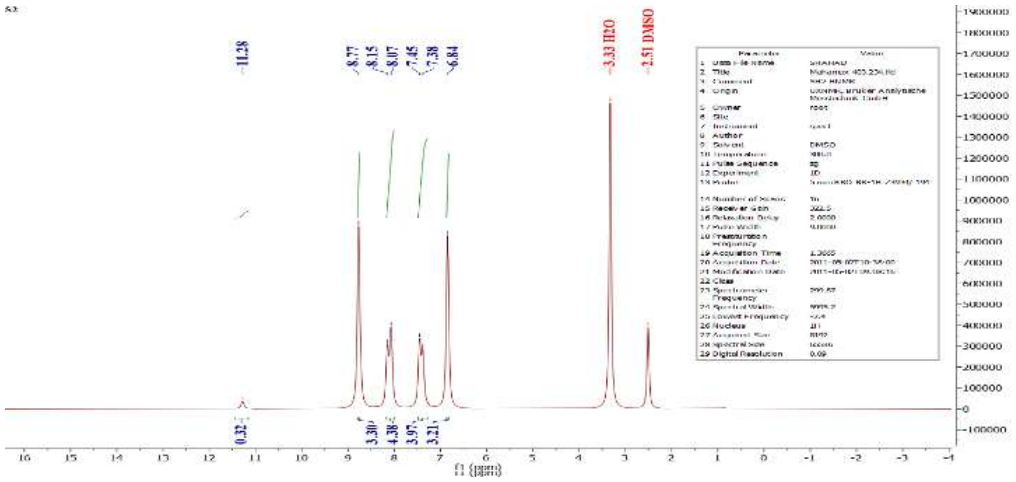


Figure 3: ^{1}H-NMR spectra of substance (S2).

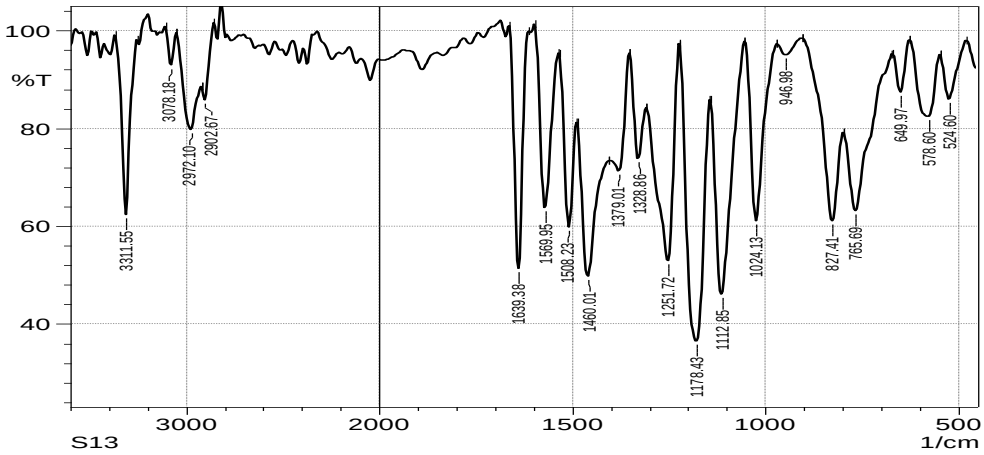


Figure 4: FT-IR spectra of coumpond (S8).

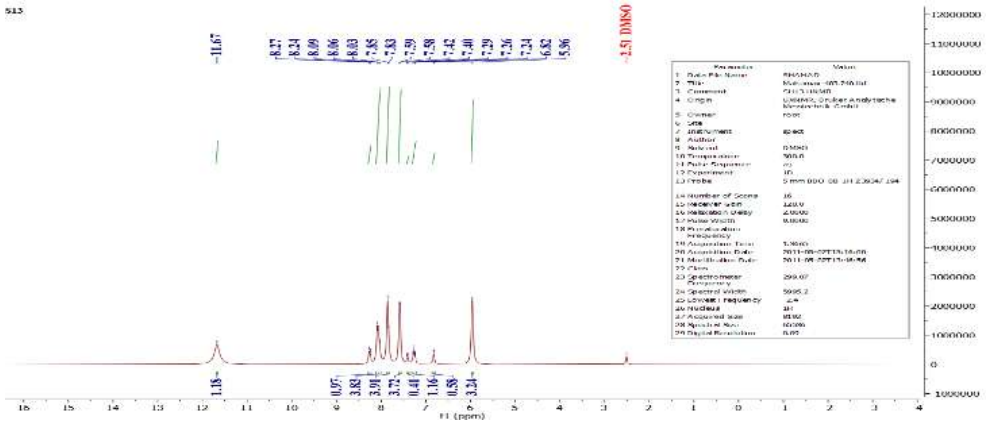


Figure 5: FT-IR spectra of coumpond (S11).

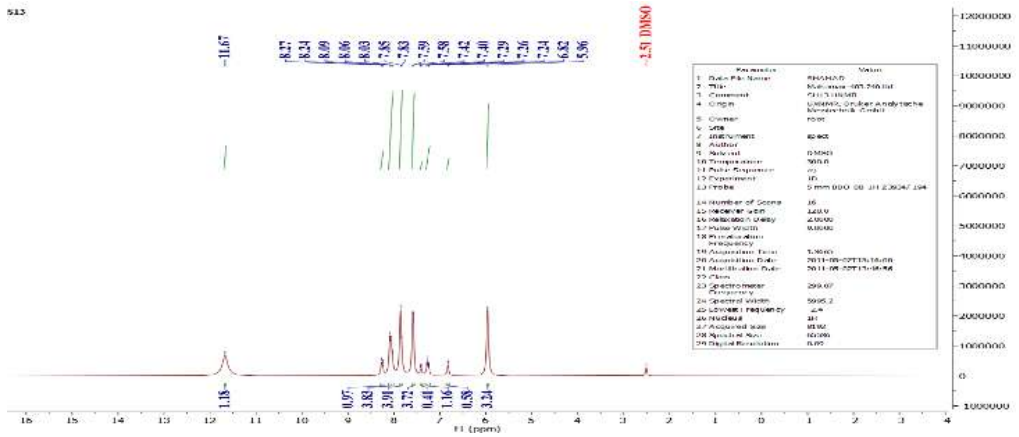


Figure 6: ^{1}H-NMR spectra of substance (S8).

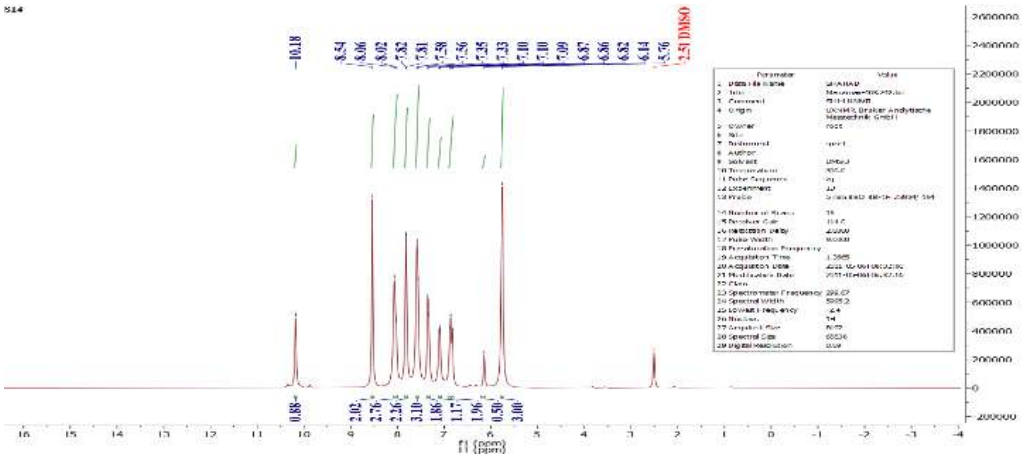


Figure 7: ^{1}H-NMR spectra of substance (S9).

Figure 8: Antibacterial activity of compund (S6) against S. Aureus.

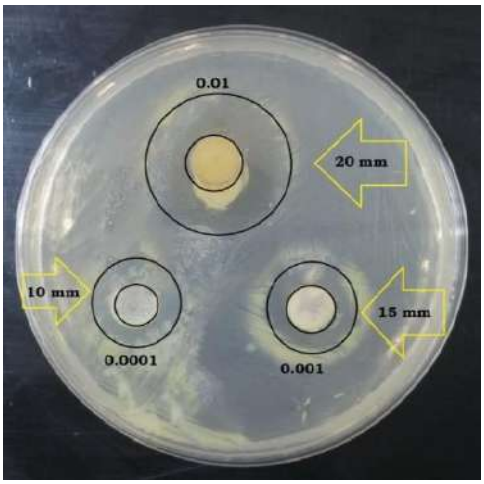


Figure 9: Antibacterial activity of compound (S9) against E. Coli.

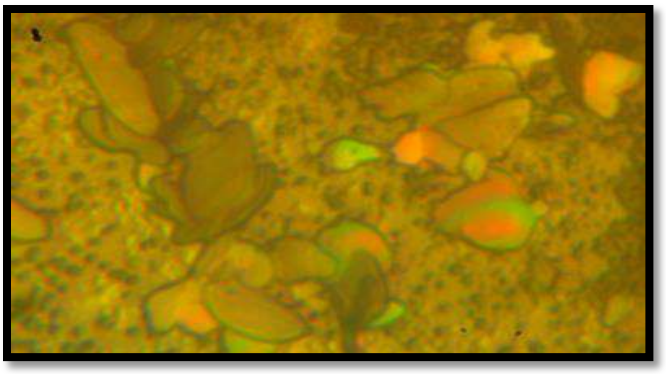
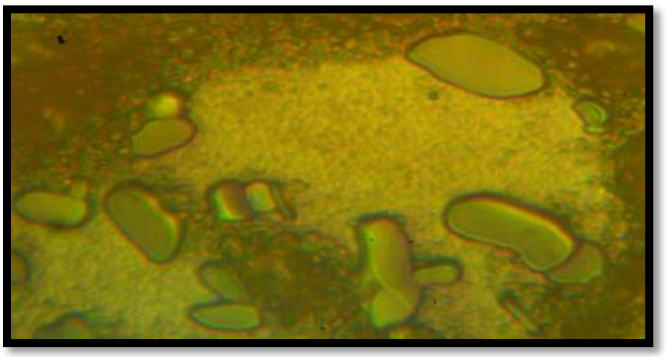
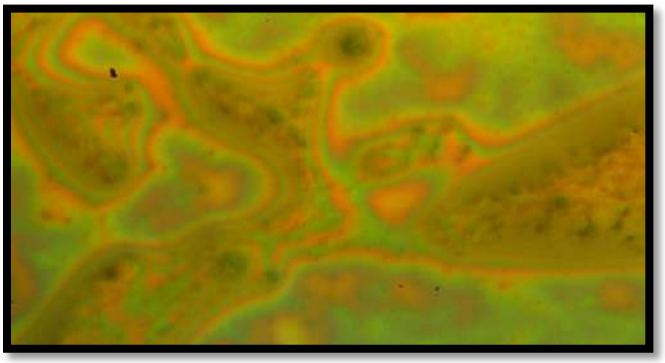


Figure 10: Textural structure of the compound



Figure(11)Textural structure of the Smectic phase of the compound[S3]



Figure(12) Nematic textural structure of the compound[S3]

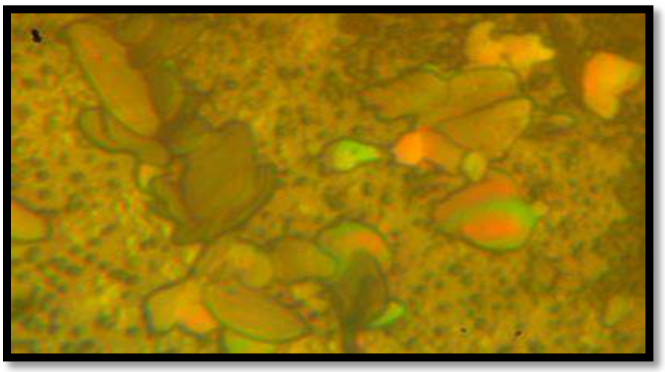


Figure (13) Nematic textural structure of the compound [S9]

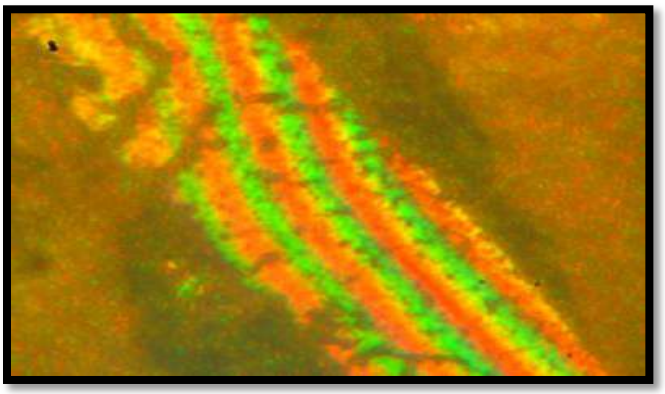


Figure (14)Nematictextural structure of the compound[S9]

4. Conclusions

Hydrazide and aldehyde replacements combine to form hydrazone, which reacts with anthralic acid to produce six rings from 2,3-dihydroquinazoline-4-one. Proton nuclear magnetic resonance and infrared spectroscopy were used to verify the legitimacy of these items. Additionally, they provided bacterial activity for the Gram stain against two different kinds of bacteria: positive and negative. Upon examining their liquid crystalline characteristics, several substances revealed thermal, Smectic, and Nematic phases.

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