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SYNTHESIS, CHARACTERIZATION, EVALUATION OF BACTERIAL ACTIVITY AND LIQUID CRYSTAL PROPERTIES OF NEW DERIVATIVES OF TETRAZOLE COMPOUNDS USING TRIETHYL BENZENE-1,3,5-TRICARBOXYLATE AS A STARTING MATERIAL

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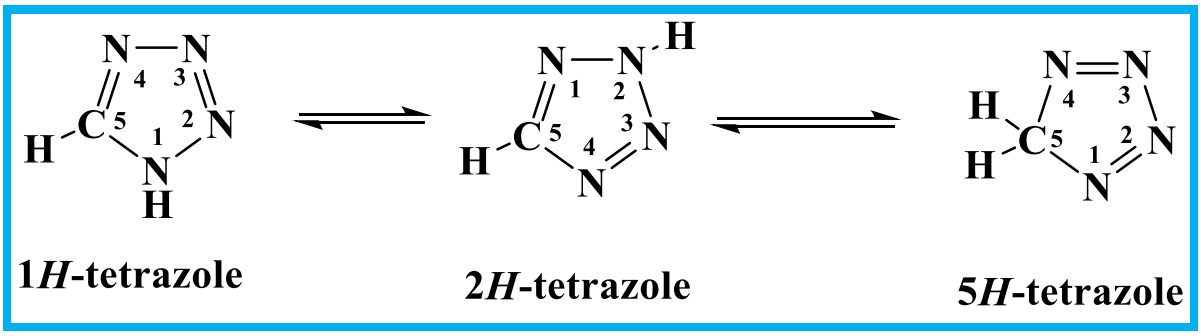
Abstract: In this research article, new derivatives of tetrazol compounds were prepared, Using physical techniques (color, melting point, flow velocity) and spectroscopic methods (infrared spectrum, nuclear magnetic resonance spectrum), this study used triethylbenzene-1,3,5-tricarboxylate as the basic nucleophile for the preparation of hydrazide by reaction with aqueous hydrazine. The latter reacted with benzaldehyde compensators to prepare derivatives of hydrozones that reacted with sodium azide to prepare pentacyclics as a final product. After confirming that the prepared compounds' compositions were accurate, the bioeffectiveness of the compound chain was examined using two different strains of Cram-positive and Cram-negative bacteria (Escherichia coli and Staphylococcus aureus). A hot polarization microscope was used to examine the liquid crystal properties and changes that occurred on some of the compounds in order to determine the phases that the compounds' crystals went through.

Keywords: Tetrazole, Liquid crystals, Biological activity.

1. Introduction

Tetrazole is a heterocyclic pentacyclic compound containing one carbonxatom and four nitrogen atoms. The molecular formula CH_2N_4[1] contains the following three isomers:



These substances are regarded as some of the most important cyclic substances. It is classified as an electron-pushing compound because it has four pairs of free electrons, which is equal to four nitrogen atoms [2]. Tetrazole compounds and their derivatives are vital in medicine, particularly in the biological field, as demonstrated by earlier research. demonstrates antifungal and antibacterial activity [3,4]. It is an effective

countermeasure for viral immunodeficiency [5], and the subsequent substances exhibit potent anticancer properties [6].

LiquidE crystalsE are substances that, at first glance, appear to be liquid. However, their particle arrangement is in distinct levels, just like that of crystals [7]. This state lies between that of a stable liquid and that of a stable crystalline solid. Some [8] propose that liquid crystals represent the fourth state of matter. Between the isotropic phase, also referred to as the liquid phase, where particle movement is unrestricted, and the solid phase, which has integrated molecular organization in both position and direction and limits particle movement, liquid crystals display a transitional state [9].

2. Materials and Methode:

2.1. Material: Without additional purification, all of the compounds utilized in this investigation were acquired from BDH, Fluka, and Aldrich.

2.2. Devices used: A thermoelectric melter 9300 was used to determine melting points. Using KBr disk at a scale of (400–4000) cm-1, Shimadzu FT-IR 8400S spectrophotometer; 1H-NMR and 13C-NMR spectra using Bruker apparatus operating at 400 MHz. Thickness at 0.2 mm, Fluka silica gel plates were employed in thin-layer chromatography (TLC). The plates were activated with fluorescent silica gel G, and visibility was achieved by UV light.

2.3.Preparation of Tetrazole (A6-A9) [10]

Mixing (mol 0.004) of prepared SHF bases (A5-A2) dissolved in (ml25) of ethanol with (MOL 0. 78g 0.012) of sodiumxazide dissolved in (ml 15) of the solvent itself, then the mixturexwas ascended for (11-15) hours, and the end of the reaction was confirmed using TLC technology, left to cool, then filtered and washed with cold water, and re-crystallized from ethanol, table (1)shows**.**

Table (1) some physical properties, percentage, reaction time and Rf of tetrazole derivatives (A6-A9).

Comp No	R	Molecular Formula	m.p. °C	Yield %	R.T Hour	R_f	Color
A_6	3,4-CH_3	$C_{36}H_{39}N_{15}O_3$ 729.81	320-322	65	15	0.99	Light yellow
A_7*	2,4-Cl	$C_{30}H_{21}Cl_6N_{15}O_3$ 852.30	300-320	69	15	0.96	Yellow
A_8*	4-OCH_3	$C_{33}H_{33}N_{15}O_6$ 735.73	302-310	63	15	0.78	Light yellow
A_9	4-OH	$C_{30}H_{27}N_{15}O_6$ 693.65	310-312	67	16	0.70	Dark yellow

2.4. Evaluation of the biological activity of:

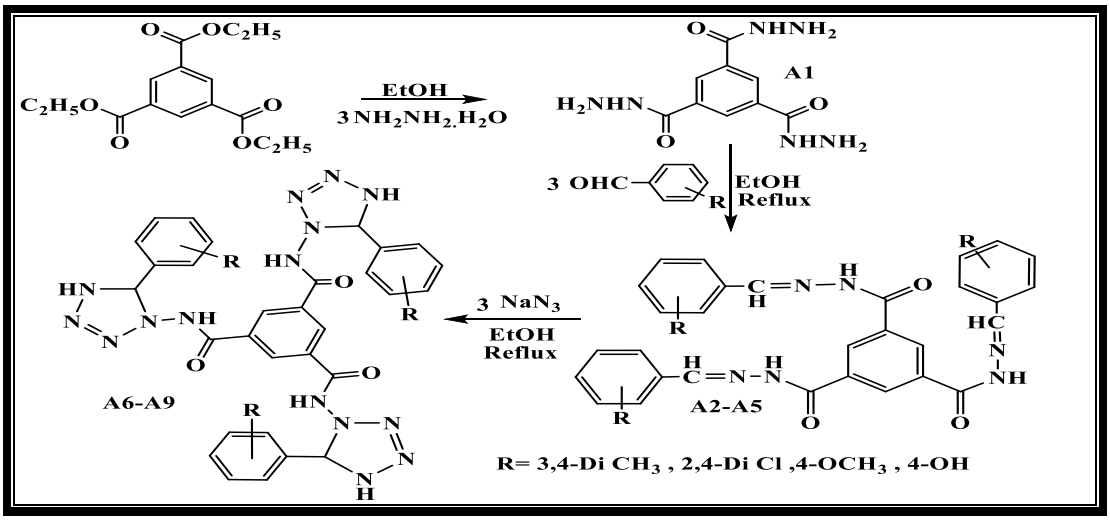
The propagation approach of the Kirby Bauer movement, which involves spreading 0.1 ml of bacterial solution on ager Muller Hinton plates and letting them absorb the fluid for five minutes, has been used to measure biological activity [11, 12]. A cork plunger and a five mm diameter cork were used to create holes in each dish. Then, 0.1 ml of the produced solutions for the fourth hole, which employed Ciprofloxacin as a reference sample, were incubated at 37 °C for twenty-four hours. Using Prescott's approach [13,14,15], the inhibitory zone widths surrounding each hole have been determined in millimeters[16,17,18].

2.5. Study ofxthe liquid crystal propertiesxof some preparedxcompounds[19]:

A hot-stage heater and polarizing microscope were used to assess the texture of some of the prepared compounds (A7, A8). MEIJI, an American company, claims to have a 38MP FHD Camera V6 with 20X magnification, an electronic thermometer to track temperature variations, and an E-PLAN 10X/0.25 160/0.17 lens. Each compound under investigation was produced on thin-film, and the model was examined closely using a magnifying glass and an automated thermal camera. Cameras were used to record the textures of the compounds that were being studied.

3. Results and Discussion:

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



Scheme (1): Path of the Ready Compounds (A1-A9)

3.1. Characterization of Tetrazole derivatives:

When studying the infrared spectrum of the prepared compounds [A9-A6], the disappearance of the beam belonging to the (C=N) Group was observed, and the appearance of two new beams that differ from the beams of derivatives of Schiff bases [A5-A2], a strong beam at the frequency (1680-1671) cm-1 belonging to the carbonyl Ester (C=O), and the appearance of a medium beam at the range (3206-3188) (3263-3322) cm-1 attributed to the appearance of other bundles at the range (1438-1419) cm-1 is attributed to the extension of the sphincter (n=n), where absorption bundles appeared at the range (3069-3063) cm-1 due to the extension of the C-H, As well as the appearance of two symmetrical and asymmetrical absorption beams respectively at the range (2843-2850) cm-1, (2935-2966) cm-1 due to the aliphatic elastic sphincter (CH), as well as the appearance of two beams at the range (1585-1570) cm-1 and (1491-1462) cm-1 due to vibration of the sphincter (C=C) aromatics[20], IR data is in Table (2) and Figure (1,2).

Table (2) results of infrared absorption (cm-1) and values of absorptions in the UV spectrum of tetrazole derivatives [A9-A6].

Comp. No.	λ max1 λ max2 EtOH nm	R	IR (KBr) cm^{-1}						
			ν (C-H) Arom.	ν N=N)(	ν C=O	ν (C=C) Arom.	ν C-H Aliph. Sym ,asy	ν(N-H)	Others
A_6	199 284	4,3-CH_3	3063	1429	1680	1585 1491	2850 2935	3198 3306	------
A_7	204 317	4,2-Cl	3069	1422	1671	1571 1479	2844 2960	3194 3278	νC-Cl 766
A_8	205 326	4-OCH_3	3066	1419	1676	1570 1462	2843 2966	3188 3263	νC-O-C Asy: 1251 sym: 1163
A_9	194 284	4- OH	3066	1438	1674	1583 1466	2649 2947	3205 3322	νOH 3434

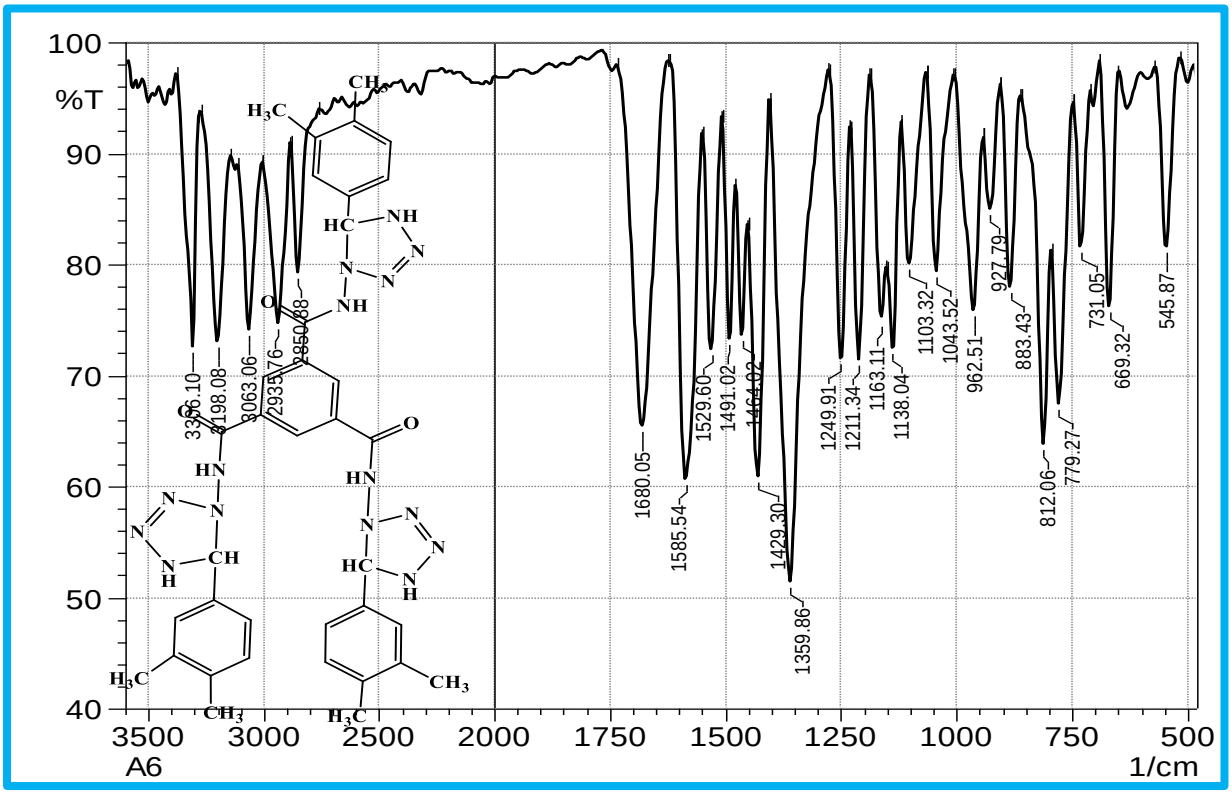


Figure (1) infrared spectrum of the compound (A6)

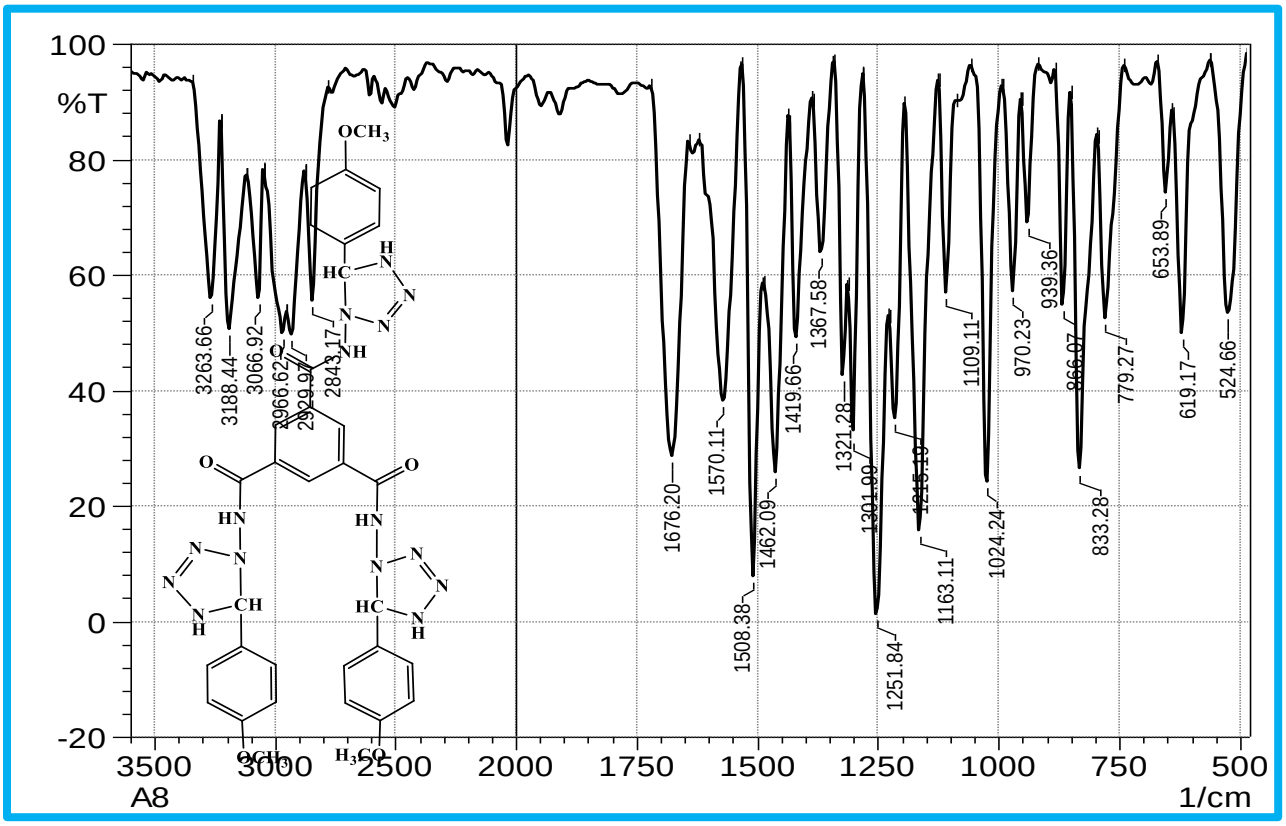


Figure (2) infrared spectrum of the compound (A8)

A single signal was seen at the site ppm (3.80) attributed to the protons of the group (CH3) and numbered (1) when examining the NMR spectrum of the proton 1H-NMR of the compound [A8] using a solvent (DMSO-d6). In between the spectrum, a single signal appeared at the site ppm (4.51) attributed to the Proton of the group (N-H) One signal was seen at the location where the pentagonal ring was formed and numbered (5); this signal, at ppm (5.60), was identified as the proton of the group (C-H). A single signal at position ppm (11.14) in the formed pentagonal ring, which was numbered (4), was identified as the proton of the amide group (N-H) and assigned the number (6), and the spectrum revealed two binary signals at the range of ppm (7.01-7.80), which were attributed to the (C-H-Ar) group numbers (3,2), respectively. Additionally, a signal at the site of ppm (8.61) was linked to the group numbered (7), while a signal at ppm (2.48) was linked to the solvent's protons (dmso-d6). As shown in Figure (3)

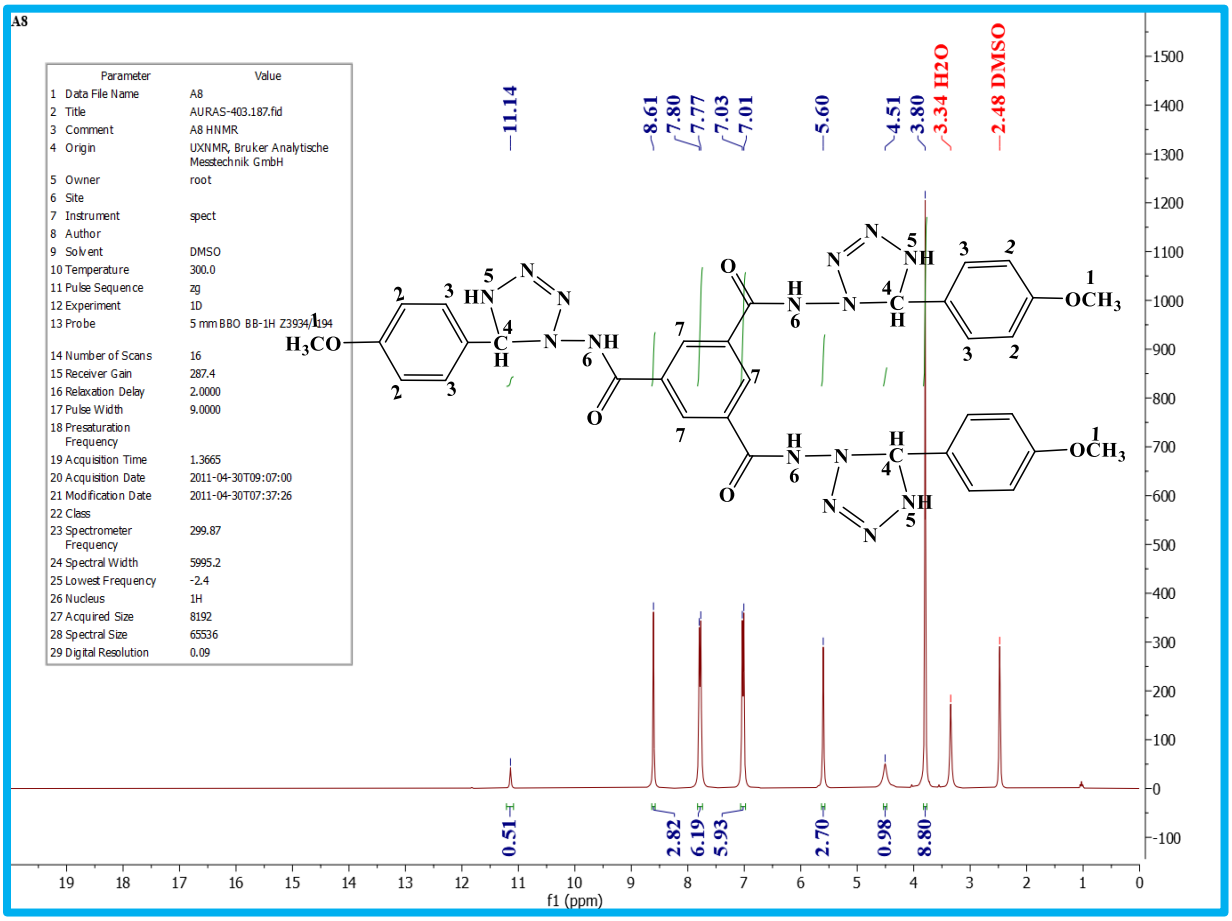


Figure (3): Proton NMR spectrum of the compound [A8]

The NMR spectrum of the compound's [A8] carbon 13C-NMR analysis revealed signals at sites ppm (55.84) attributed to the group (CH3) numbered (1) and ppm (97.42) belonging to the Group (C-H) numbered (6) in the formed tetrazole ring. Additionally, the spectrum revealed signals at sites (160.99,137.99,130.40,127.02,122.46,114.84) ppm belonging to the carbon atoms of aromatic rings (C-H-Ar) numbered (2,5,9,8,4,3), respectively. A signal from the carbon atom group (C=O) with number (6) emerged in the spectrum at ppm (165.12). in addition to the appearance of a multiple signal at the range (40.78-39.11) ppm belonging to the solvent carbon atoms (dmso-d6). As in the Figure (4)

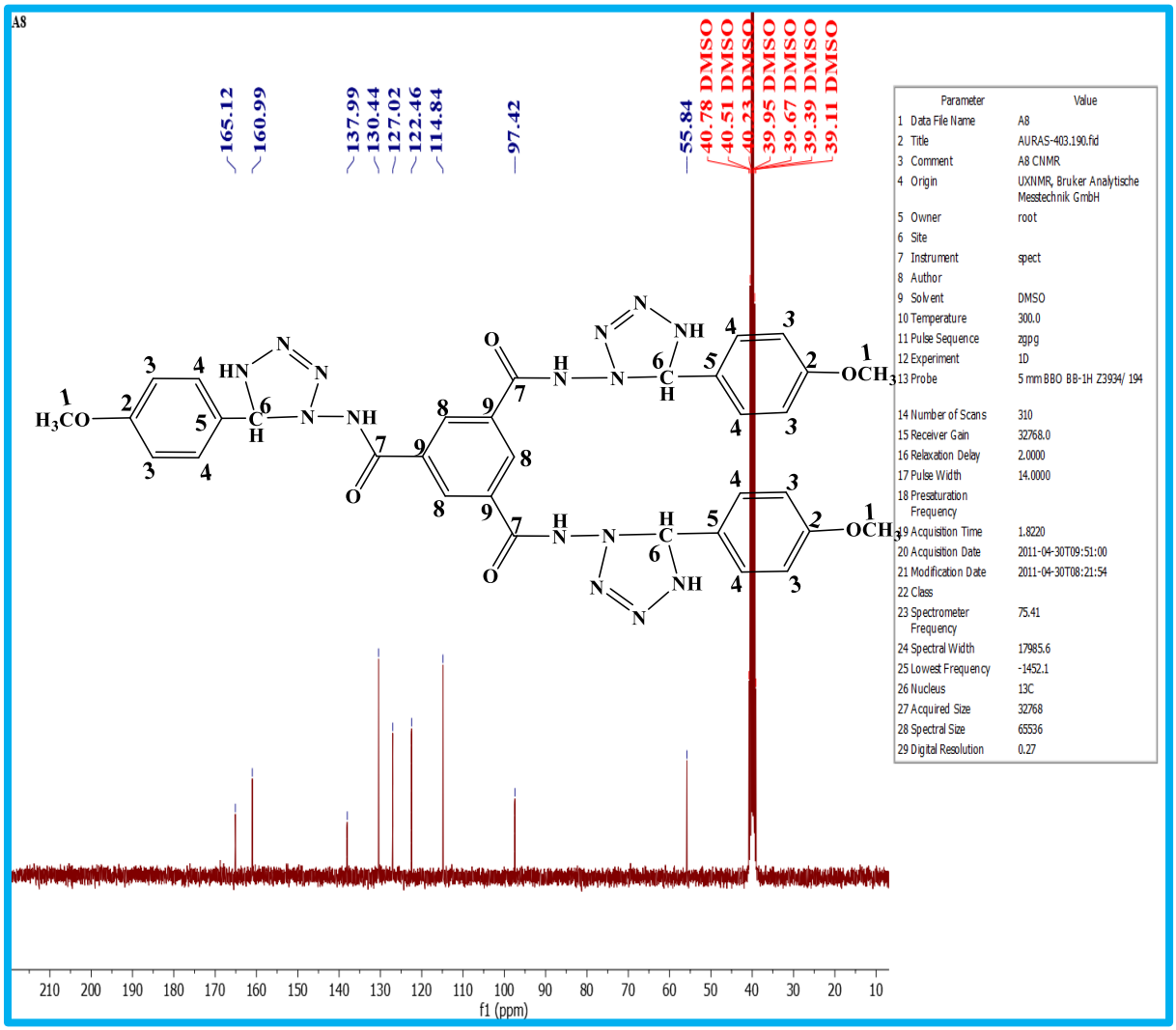


Figure (4): carbon NMR spectrum of the compound [A8]

3.4. Evaluation of Biological activity of (A6, A7, A8)

Initially, the gram-positive bacteria Staphylococcus aureus and the gram-negative coliform bacteria E. coli were the two pathogenic bacterial species against which the agar diffusion method conducted biological activity[21, 22]. The substances had biological effects [23,24, 25]. as shown in the following table 3

Table (3): Antibiotics and other generated chemicals can prevent the growth

Comp. No.	E. Coil Conc. mg/ml			Staph. Aureus Conc. mg/ml		
	0.0001	0.001	0.01	0.0001	0.001	0.01
A6	15	12	0	20	17	14
A7	0	0	0	17	15	10
A8	15	10	0	15	15	12
Amoxicillin	20	20	10	24	16	10

3.5. DiscussionXand identification of liquidXcrystal phases[26]:

Tables (4) illustrate how the thermal transition degrees of the liquid and isotropic crystalline phases of the majority of the prepared compounds were determined, their nature examined, and liquid crystal forms identified with a polarized light microscope and heater.

Table (4): liquid crystal phase transitions in a device Mic.sc for prepared vehicles

Apparatus	NO.	Crystal	Smectic A	Smectic C	Nematic	ΔT_{SA}	ΔT_{SC}	ΔT_{N}
Microscope	A7	300	320	-------	-------	20		
	A8	302	310	--------	-------	8		

Due to the presence of chlorine (Cl) atoms at the positions 4,2 - dichloro (2,4-dichlo) as terminal aggregates, which are distinguished by a strong dipole moment that increases the ratio of peripheral to lateral attractive forces, compound [A7] only displayed the Smectic phase with a high thermal range. similar to Figure (5).

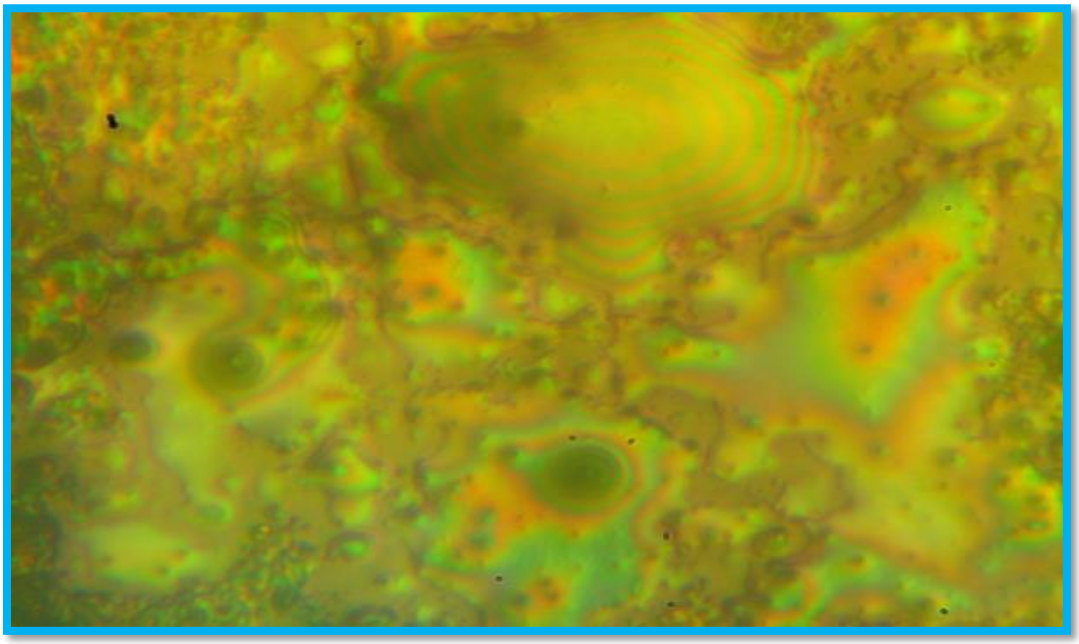


Figure (5) histological structure Smectic ladder compound[A7]

Due to the presence of the methoxy group (OCH3) at the 4-methoxy site, the compound [A8] only displayed the Smectic phase with a high temperature range. Terminal aggregates, on the other hand, are defined as electron-propelling aggregates that increase the ratio of peripheral to lateral attractive forces.similar to Figure (6).

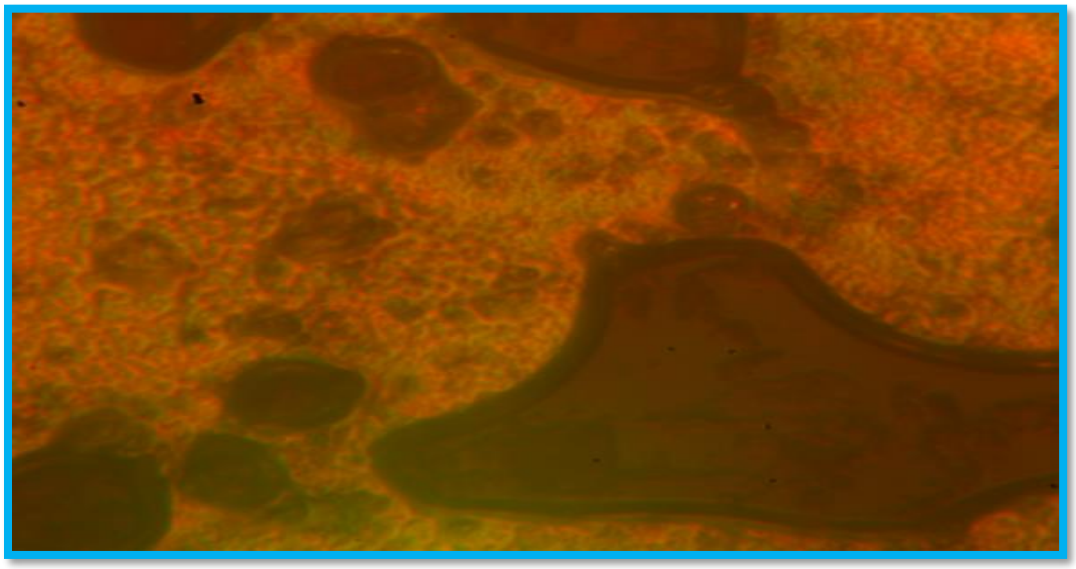


Figure (6) histological structure Smectic ladder compound[A8]

Conclusion:

The correctness and validity of the produced compounds were confirmed by spectroscopic and physical studies. For instance, increased proton and carbon nuclear magnetic resonance spectra confirmed the presence of active aggregates with great precision, and these spectra also agreed with the structures of the produced compounds. These spectra were obtained using infrared and ultraviolet spectroscopy. These materials are stable at lab temperature; they neither deteriorate nor change color. The produced compounds showed potent and efficient inhibitory activity against both gram-positive and gram-negative bacteria when compared to control samples that contained antibiotics. Furthermore, analysis of the compounds' liquid crystals in the Smectic (S) phases demonstrated proof of the system's consecutive, polarized terminal groups.

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