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Synthesis, and Characterization of Pentacyclic Thiazolidinedione

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Abstract: This study involves the synthesis of Pentacyclic Thiazolidinedione Derivatives (B1-B7) through the reaction of 1 mole of Schiff bases with 1 mole of thioglycolic acid, dissolved in absolute ethanol. The validity of the compound structures was confirmed using physical and spectroscopic methods such as infrared spectroscopy, proton nuclear magnetic resonance spectroscopy, and Mass spectrum. Additionally, melting points and purity were determined, and reaction progress was monitored by Thin-Layer Chromatography (TLC).

Key points: Pentacyclic, Thiazolidinedione, Schiff bases, Thioglycolic acid.

1. Introduction

Thiazolidinone pentagons represent a class of organic compounds with a unique molecular architecture and versatile chemical properties. Derived from the parent compound thiazolidinone, which itself is a heterocyclic organic compound containing sulfur and nitrogen atoms in its five-membered ring structure [1], thiazolidinone pentagons exhibit a remarkable array of biological activities and synthetic applications [2]. This introduction delves into the structural features, synthetic methods, biological significance, and potential applications of thiazolidinone pentagons, elucidating their importance in medicinal chemistry, materials science, and beyond [4]. Thiazolidinone pentagons are characterized by a central five-membered ring containing atoms of carbon, sulfur, and nitrogen [5]. This core structure imparts unique physicochemical properties to these compounds, making them attractive targets for synthetic chemists and pharmaceutical researchers. The flexibility of thiazolidinone pentagons allows for diverse functionalization at various positions around the ring, leading to the synthesis of a wide range of derivatives with tailored properties [6]. The synthesis of thiazolidinone pentagons encompasses a variety of strategies [7], each tailored to achieve specific structural modifications and functional group manipulations [8,9]. Classical approaches often involve the condensation of thioamides with α-halo carbonyl compounds, followed by cyclization to form the thiazolidinone ring. Alternatively, multicomponent reactions and transition-metal-catalyzed processes have emerged as efficient routes for accessing diverse thiazolidinone pentagon scaffolds with high regio- and stereo-selectivity [10,11]. Thiazolidinone pentagons exhibit a broad spectrum of biological activities, rendering them promising candidates for drug discovery and development [12]. Their pharmacological profiles encompass anti-inflammatory, antibacterial, antiviral, anticancer, antidiabetic, and antifungal properties, among others [13]. The ability of thiazolidinone pentagons to modulate various molecular targets within biological systems underscores their potential as therapeutic agents for the treatment of a wide range of diseases [14]. Beyond their pharmaceutical relevance, thiazolidinone pentagons find applications in diverse fields such as materials science, agrochemicals, and catalysis [15,16]. Their structural diversity, combined with their tunable physicochemical properties, makes them valuable building blocks for the design and synthesis of functional materials, including polymers, liquid crystals, and sensors [17]. Moreover, the catalytic activity of certain thiazolidinone pentagon derivatives has been explored in asymmetric synthesis and other organic transformations,

highlighting their utility in synthetic chemistry [18]. Thiazolidinone pentagons represent a fascinating class of compounds with significant implications in both fundamental research and practical applications [19]. Their structural versatility, synthetic accessibility, biological activities, and potential applications position them as pivotal players in the ever-expanding landscape of organic chemistry and interdisciplinary science [20].

2. Experimental:

2.1. Material and Devices used: The chemicals materials are collected from Fluka, Aldrich, and BDH and used without further purification. The melting points were measured using Electrothermal Melting Apparatus 9300. Bruker FT-IR 8400S spectrophotometer with a scale of (400-4000) cm^{-1} by KBr disc. ^{1}H-NMR spectra on Bruker instruments running at 400 MH_Z. Thin Layer Chromatography (TLC) was performed using Fluka silica gel plates with 0.2 mm thickness, activated with fluorescent silica gel G, and visualization was achieved using UV light.

2.3. Synthesis of Pentacyclic Thiazolidinedione Derivatives (B1-B7)

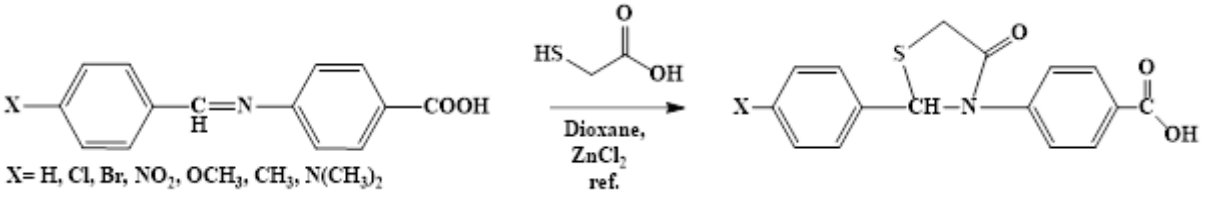
0.02 moles of sodium hydroxide were dissolved in 25 ml of dioxane and heated with stirring for ten minutes in a 100 ml round-bottom flask equipped with a condenser and immersed in a water bath. Then, 0.02 moles (2.7 gm) of zinc chloride anhydrous dissolved in 10 ml of dioxane were added as a catalyst to 0.02 moles (2 gm) of thioglycolic acid dissolved in 20 ml of dioxane. The mixture was then added dropwise, and upon completion of mixing, immediate turbidity of the mixture was observed. Stirring and refluxing of the reaction mixture continued for 18-22 hours. The progress of the reactions was monitored using thin-layer chromatography with a benzene: methanol (6:4) solvent system and visualization with iodine. After completion of the reflux period, a portion of the solvent was evaporated, yielding a residue. The residue was filtered, washed with distilled water, dried, then washed again with chloroform [21,22]. Table (1) summarizes some of the physical properties and percentage yields of compounds B1-B7 prepared.

Table (1): Yield ratios and some physical properties of compounds (B1-B7).

Comp.	X	Molecular Formula	Mol.wt. gm/mole	Yields %	R*f*	M.P. ^{O}C	Color
B_1	H	$C_{54}H_{39}N_3O_9S_3$	299.34	70	0.70	245-247	Yellow
B_2	Cl	$C_{54}H_{36}Cl_3N_3O_9S_3$	333.79	76	0.78	229-231	light Orange
B_3	Br	$C_{54}H_{36}Br_3N_3O_9S_3$	378.24	72	0.80	212-214	Reddish orange
B_4	NO_2	$C_{54}H_{36}N_6O_{15}S_3$	344.34	65	0.75	236-238	Reddish orange
B_5	OCH_3	$C_{57}H_{45}N_3O_{12}S_3$	329.37	80	0.83	223-225	Yellow
B_6	CH_3	$C_{57}H_{45}N_3O_9S_3$	313.37	84	0.79	216-218	Yellow
B_7	$N(CH_3)_2$	C18H18N2O3S	342.41	82	0.81	235-237	Yellow

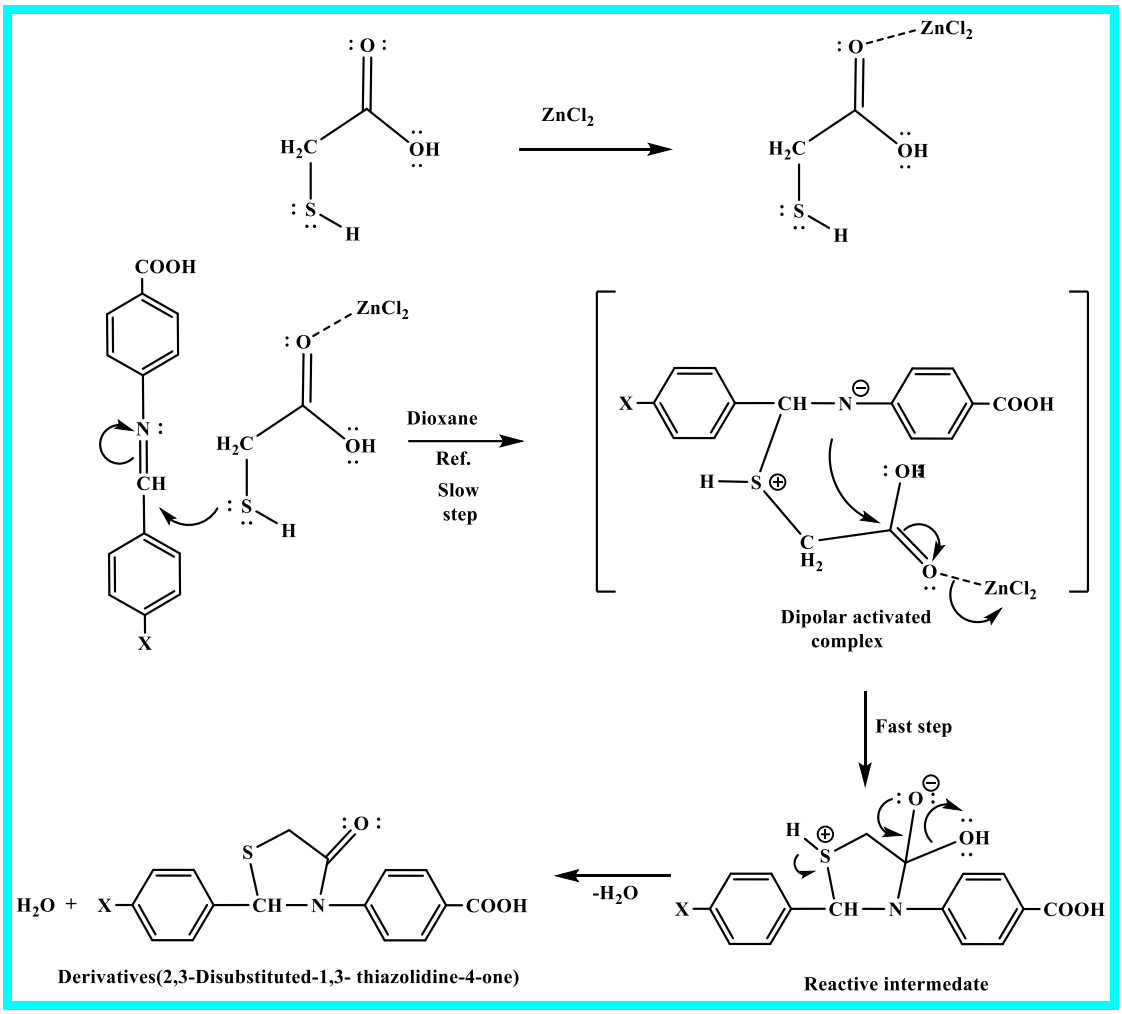
3. Results and Discussion

Pentacyclic Thiazolidinedione Derivatives B1-B7 synthesis by 1 mole of Schiff base with 1 mole thioglycolic acid, as shown in Scheme 1.



Scheme (1): Structural formula of the prepared compounds (B1-B7)

Substituted 1,3-thiazolidine-4-one derivatives were synthesized via the reaction of schiff bases derivatives with thioglycolic acid using reflux conditions and dioxane as the solvent in the presence of zinc chloride anhydrous as a catalyst. The progress of the reactions was monitored, and reaction times were determined using thin-layer chromatography by comparing the flow rates of the resulting material with the flow rates of schiff bases derivatives, followed by visualization with iodine [23,24]. Literature suggests that the mechanism of preparation of pentagonal ring compound derivatives is heterogeneous, involving a nucleophilic attack by the sulfur atom of thioglycolic acid on the electrophilic carbon atom of the azomethine group, followed by another nucleophilic attack by the nitrogen atom of the azomethine group on the electrophilic carbon atom of the carboxyl group in thioglycolic acid [25,26]. The following mechanism illustrates the preparation of these compounds according to Scheme (2):



Scheme (2): Mechanism of Preparation of Substituted 1,3-Thiazolidine-4-one Derivatives.

3.1. Characterization of compounds (B1-B7) by FT-IR

The prepared compounds were spectroscopically characterized using FT-IR spectroscopy, and the spectra of compounds (B1-B7) indicated the disappearance of the vibrational bands of the azomethine group (νC=N) of the schiff bases prepared as the first step, which showed absorption bands within the range of cm^{-1} (1622-1636) [27]. The compounds (C5-C1) showed absorption bands of the vibrational stretching of the carboxy group (νO-H carboxy) within the range of cm^{-1} (2542-3268), with the appearance of vibrational absorption bands (νC-H arom) within the range of cm^{-1} (3058-3110), and the appearance of vibrational absorption bands of the lactam carbonyl group (νC=Olactam) within the range of cm^{-1} (1678-1699), as well as the appearance of vibrational absorption bands of the carboxyl carbonyl group (νC=Ocarboxy) within the range of cm^{-1} (1699-1655) [28]. Additionally, the vibrational absorption bands of the (ν(CO)-N) group appeared within the range of cm^{-1} (1413-1515), and the appearance of the vibrational absorption bands of the (νC-N)

group ranged from cm^{-1} (1252-1288). Furthermore, the appearance of the vibrational absorption bands of the (νC-S) group ranged from cm^{-1} (683-693) [29]. The appearance of these bands is not considered conclusive evidence of compound preparation but is preliminary evidence of the validity of the method used in preparing these compounds [30, 31]. Table (2) illustrates the absorption band values of the FT-IR spectra of compounds (B1-B7) prepared:

Table (2): FT-IR absorption results for Compounds (B1-B7) Measured in cm^{-1}.

Comp. Symb.	-X	νOH	νC-H			νC=O lactam	νC=O Carboxy	ν C=C ring	
			Arom.	Aliph. Asym. & Sym.					
B_1	-H	3221-2545	3058	2978	2901	1678	1678	1596	1413
B_2	-Cl	3221-2551	3130	2979	2902	1685	1685	1590	1487
B_3	-Br	3262-2549	3059	2980	2906	1694	1655	1579	1485
B_4	$-NO_2$	3259-2542	3068	2973	2926	1683	1683	1594	1515
B_5	$-OCH_3$	3268-2554	3064	2985	2911	1699	1660	1584	1490
B 6	$-CH_3$	3228-2542	3076	2982	2908	1681	1659	1579	1496
B 7	$-N(CH_3)_2$	3221-2552	3062	2979	2905	1679	1629	1581	1493

3.2. Characterization of compounds (B3 B4) by ^{1}H-NMR

The structural formulas of the compounds were confirmed using proton nuclear magnetic resonance spectroscopy (^{1}H-NMR). The spectrum of compound (B3) exhibited a singlet signal at a chemical shift [δ=(12.71) ppm, (s,1H), COOH] attributed to the proton of the hydroxyl group in the carboxylic acid. Additionally, a multiplet signal appeared at [δ=6.77-8.51 ppm, (m,8H) H-Ar.] corresponding to the protons of the aromatic rings. Furthermore, a singlet signal at a chemical shift [δ=(6.52) ppm, (s,1H), CH thiazolidine] was observed, attributed to the proton of the thiazolidine group, along with another singlet signal at [δ=(4.08) ppm, (s,2H), CH2 thiazolidine] corresponding to the two protons of the thiazolidine group. Finally, a singlet signal at [δ=(3.87) ppm, (s,3H), CH3 methoxy] was detected, attributed to the proton of the methoxy group [32, 33]], as illustrated in Figure (6). Similarly, the spectrum of compound (B4) displayed a singlet signal at a chemical shift [δ=(12.80) ppm, (s,1H), COOH] attributed to the proton of the hydroxyl group in the carboxylic acid. Moreover, a multiplet signal appeared at [δ=7.58-8.74 ppm, (m,8H) H-Ar.] corresponding to the protons of the aromatic rings. Additionally, a singlet signal at [δ=(6.64) ppm, (s,1H), CH thiazolidine] was observed, attributed to the proton of the thiazolidine group, along with another singlet signal at [δ=(4.21) ppm, (s,2H), CH2 thiazolidine] corresponding to the two protons of the thiazolidine group [34, 35]. These findings are depicted in Figure (7).

Table (2): Chemical Shifts of Compounds (B3, B4) Identified by Proton Nuclear Magnetic Resonance Spectroscopy Measured in ppm.

Comp. Symb.	Chemical Shift(ppm)	No. of Protons	Type of single	Group
B3	3.87	3	s	CH3 methoxy
	4.08	2	s	CH2 thiazolidine
	6.52	1	s	CH thiazolidine
	6.77-8.51	8	m	Ar-H

	12.71	**1**	**s**	**-OH Carbox.**
B4	**4.21**	**2**	**s**	CH2 thiazolidine
	6.64	**1**	**s**	CH thiazolidine
	7.58-8.74	**8**	**m**	**Ar-H**
	12.80	**1**	**s**	**-OH Carbox.**

3.2. Characterization of compounds (B3, B4) by Mass

To validate the structures of the prepared compounds, a selection of them was subjected to mass analysis to determine their molecular weights. This was achieved by identifying the molecular ion peak and other peaks corresponding to fragment ions, as well as the base peak of the molecular ion after ionization. These details are illustrated in the fragmentation pattern of each spectrum [36, 37]. As in shown figures (7,8) and schemes (3,4).

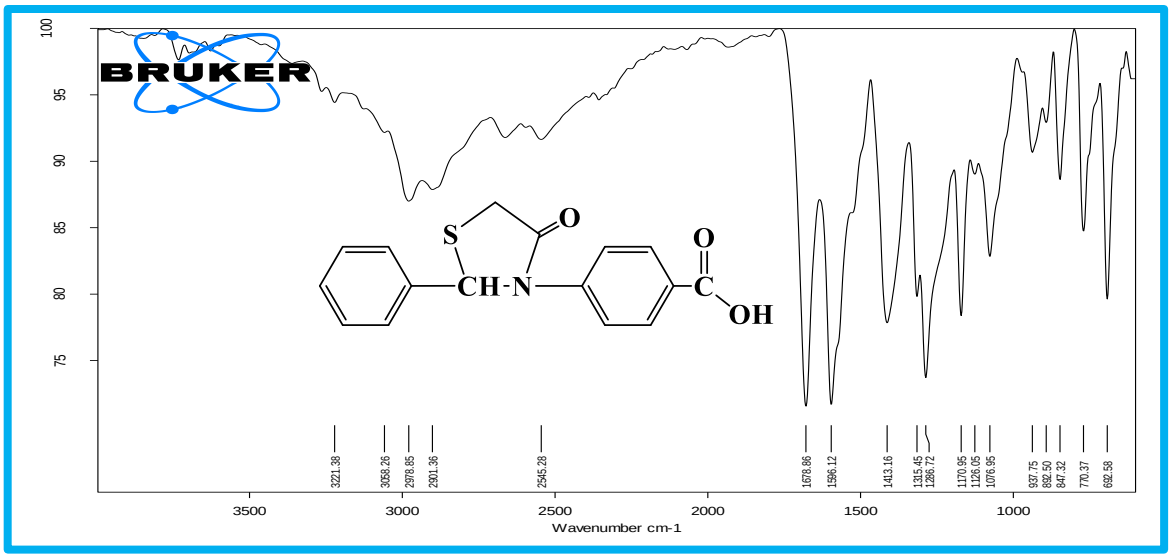


Figure (1): FT-IR spectrum of the compound (B1).

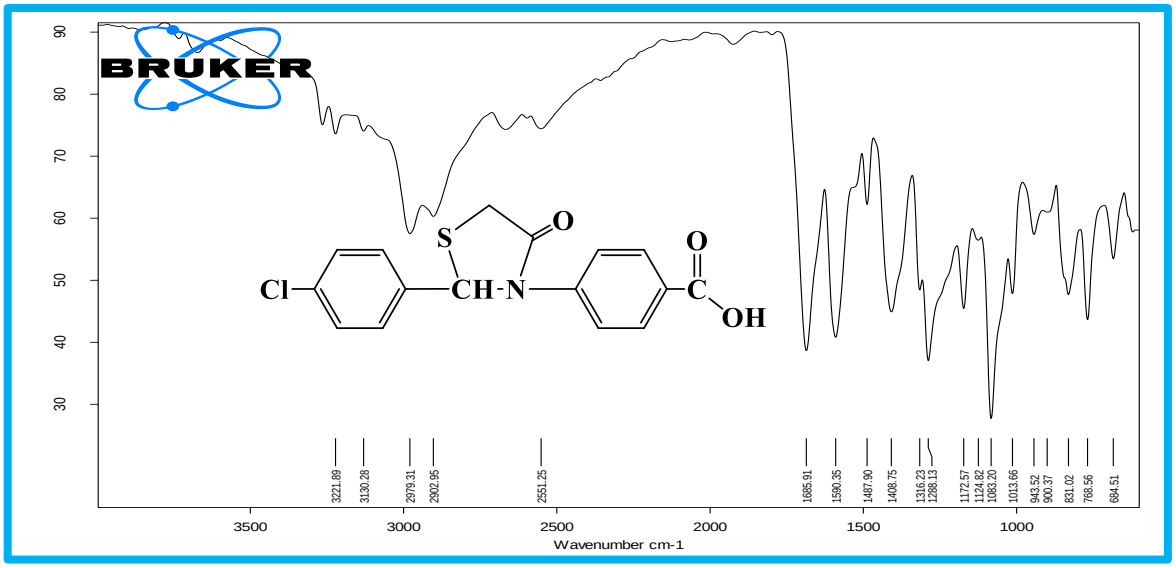


Figure (2): FT-IR spectrum of the compound (B2).

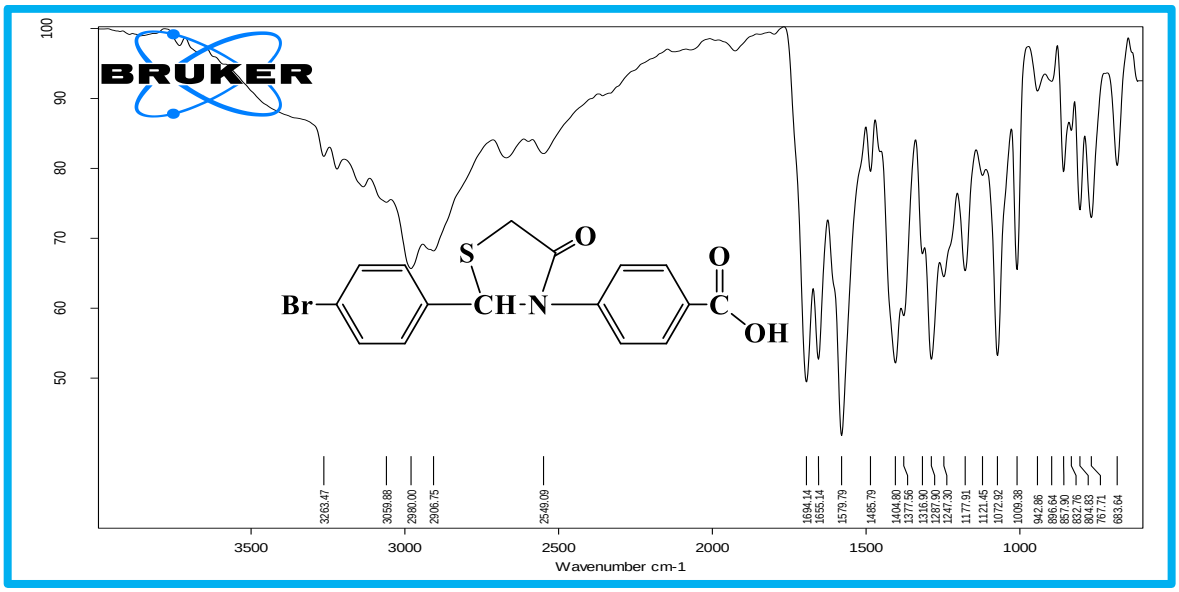


Figure (3): FT-IR spectrum of the compound (B4).

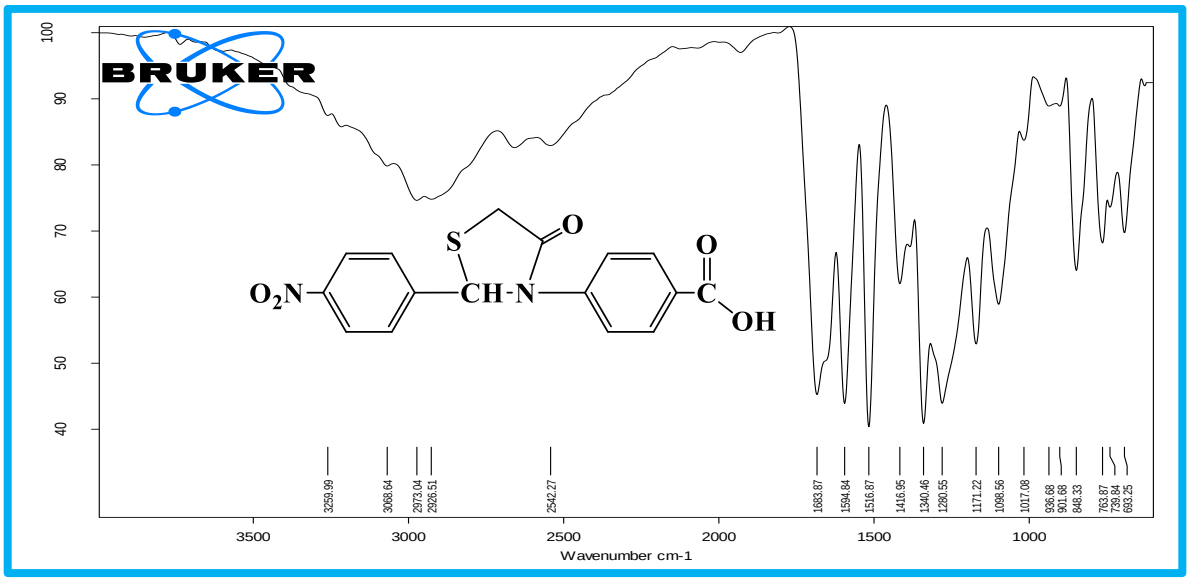


Figure (4): FT-IR spectrum of the compound (B4).

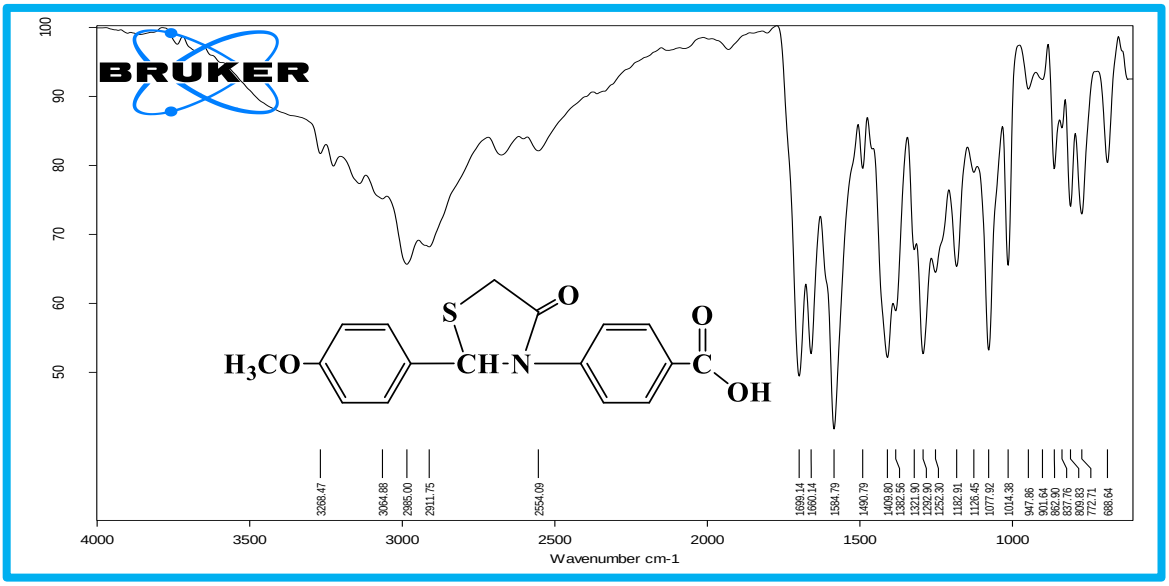


Figure (5): FT-IR spectrum of the compound (B5).

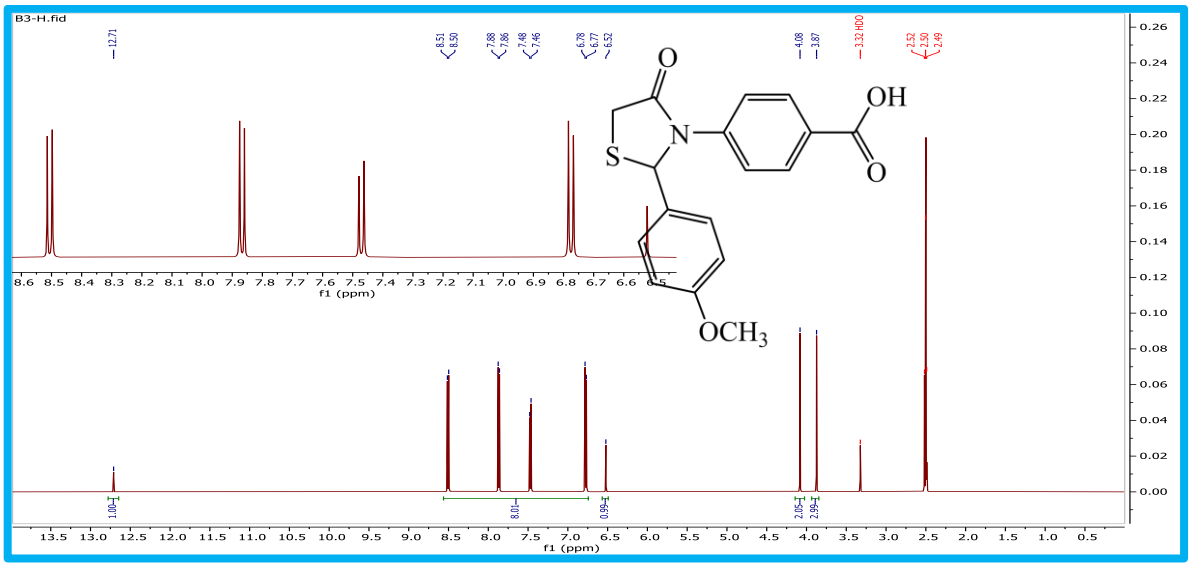


Figure (6): ^{1}H-NMR spectrum of the compound (B3).

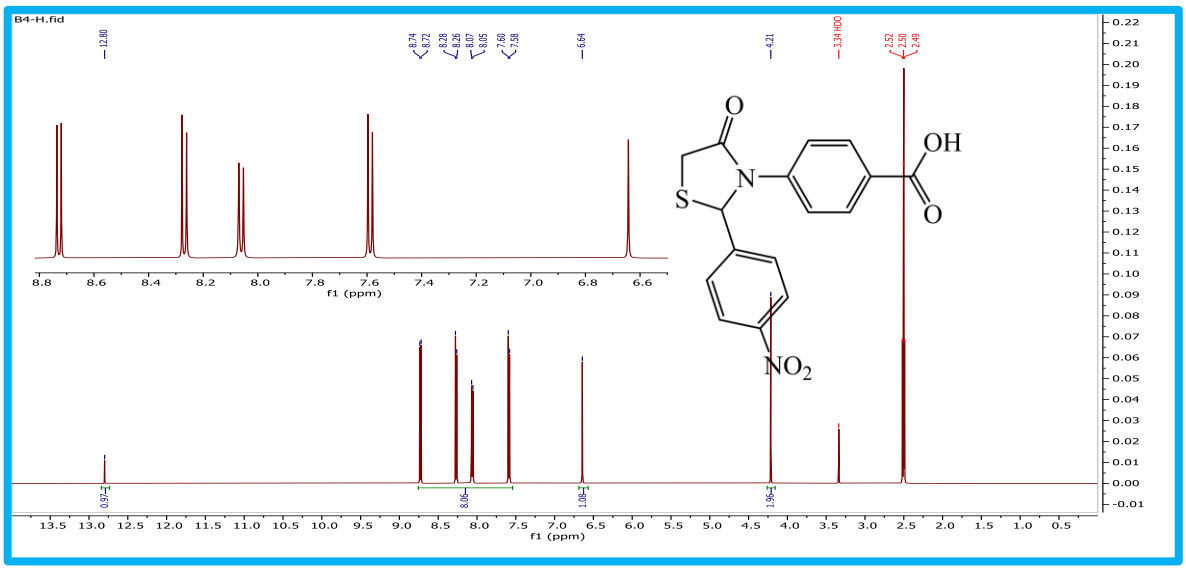


Figure (7): ^{1}H-NMR spectrum of the compound (B4).

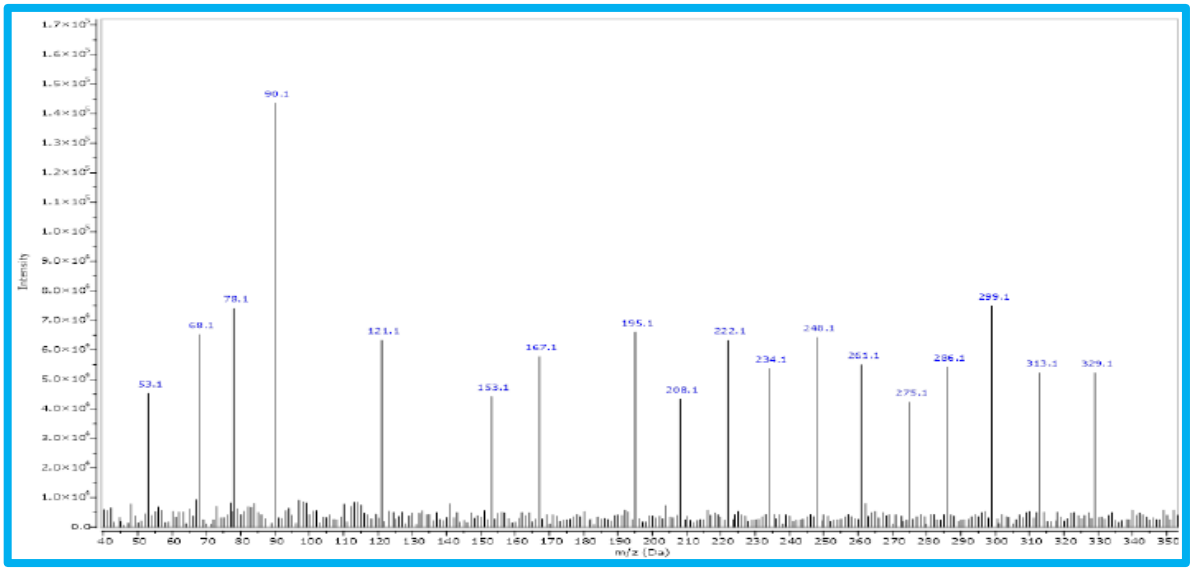
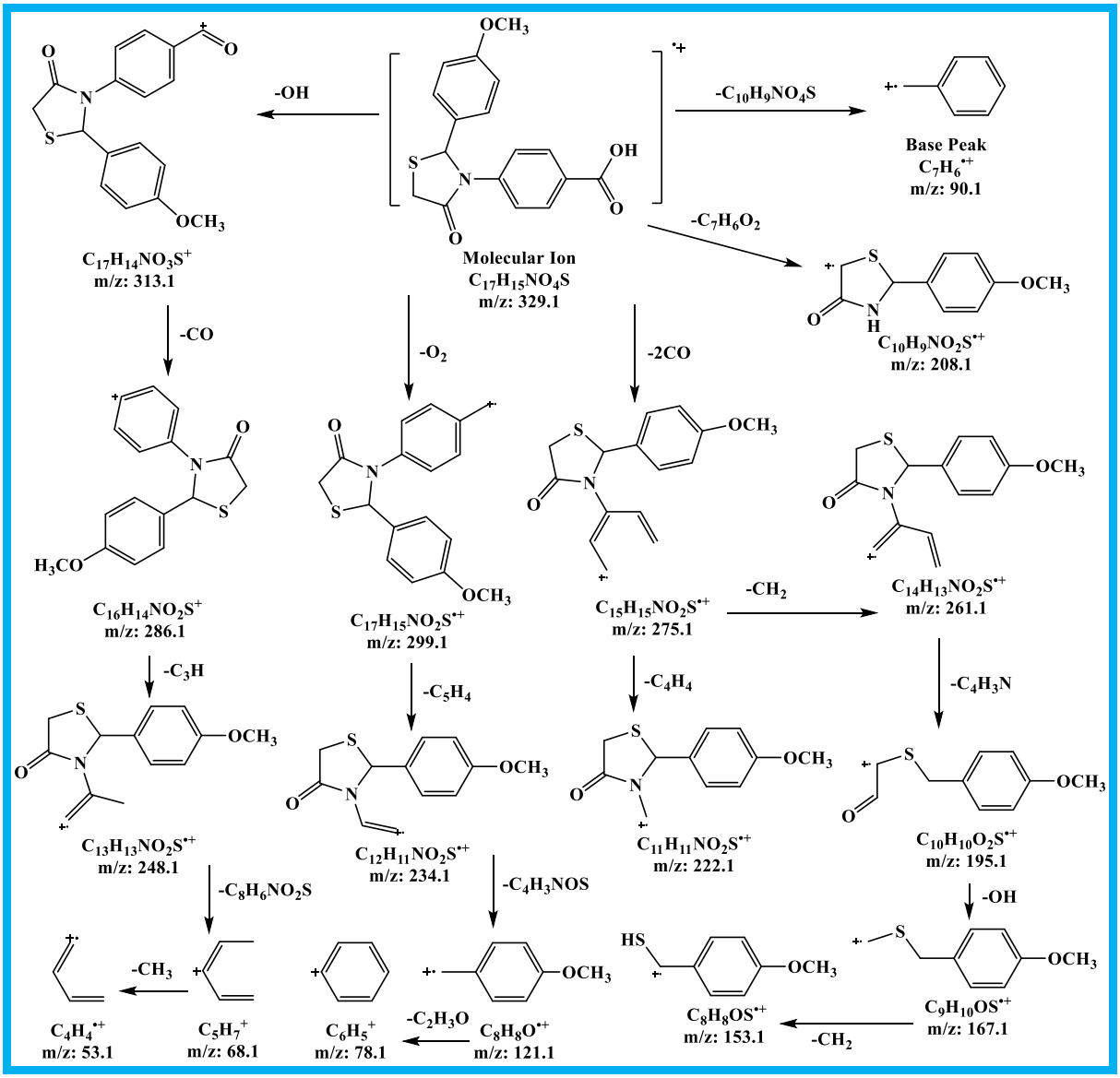


Figure (8): Mass spectrum of the compound (B3).



Scheme (3): Fragmentation pattern of compound (B3).

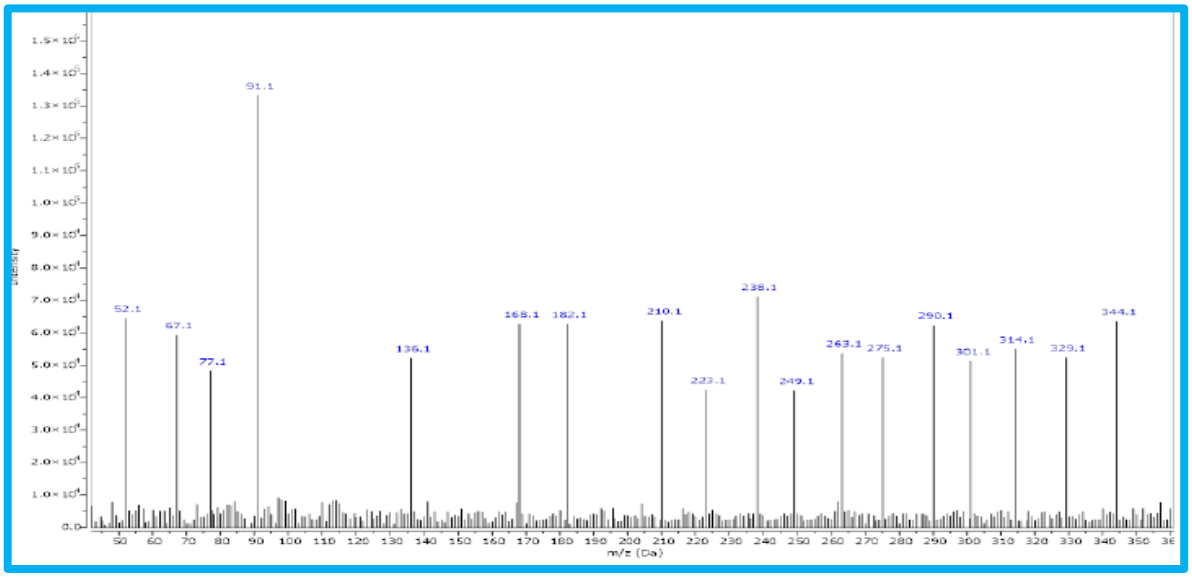
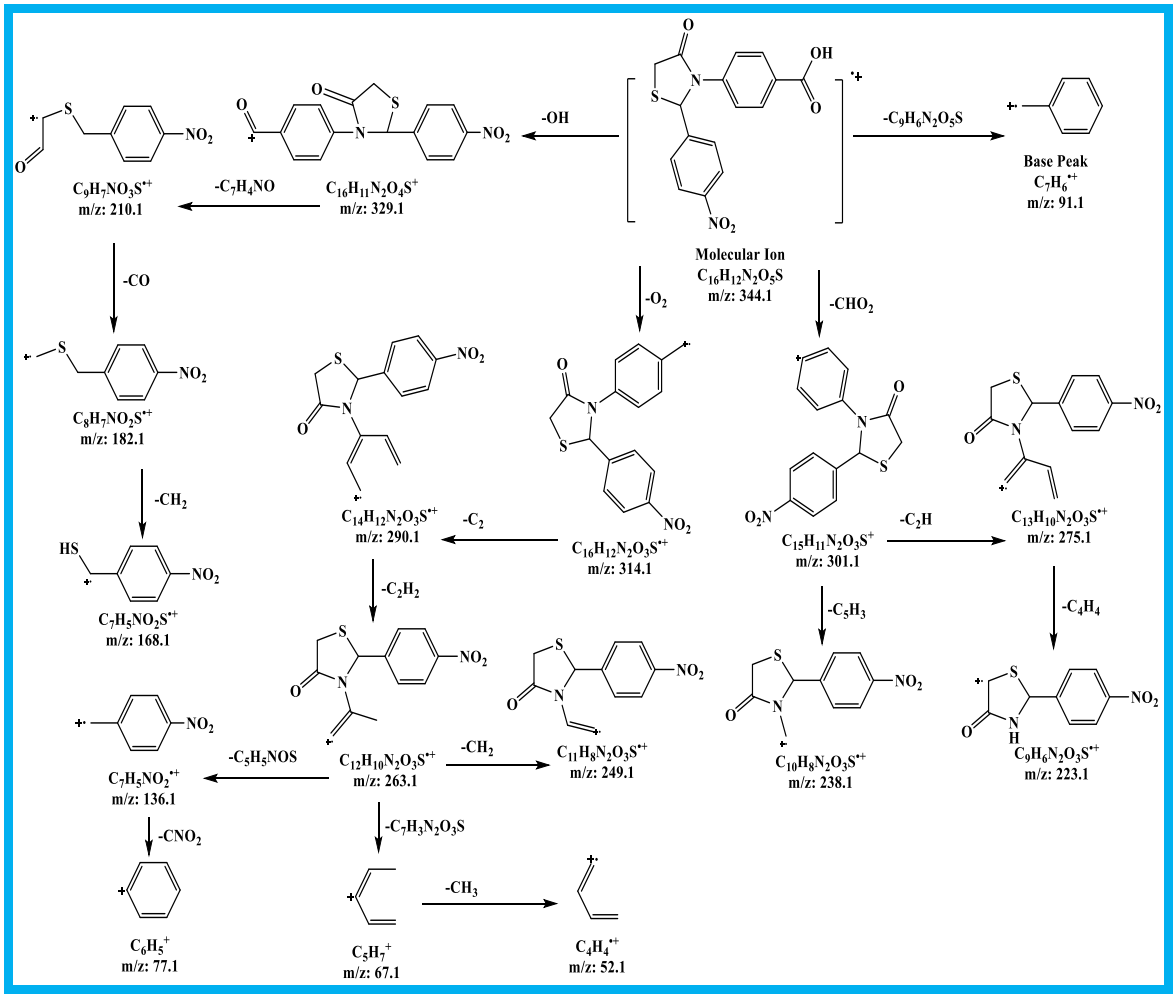


Figure (9): Mass spectrum of the compound (B4).



Scheme (4): Fragmentation pattern of compound (B4).

4. Conclusions

Physical and spectroscopic measurements have proven the accuracy of the prepared compound formulations, and that the preparation method was a successful, low-cost method with a high yield rate, so it is considered an economical preparation method.

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