

SYNTHESIS OF NEW HETEROCYCLIC COMPOUNDS DERIVED FROM 3-CHLOROBENZO[B]THIOPHENE-2-CARBONYL CHLORIDE

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ABSTRACT

In this work twenty-three compounds derived from cinnamic acid were synthesized. Acid chloride(C1) was synthesized by the reaction of cinnamic acid and thionyl chloride. Carbohydrazide (C2) was obtained by the reaction of 3-Chlorobenzo[b]thiophene-2-carbonyl chloride (C1)with hydrazine hydrate. Compound(C2) undergoes the character condensation reaction with different aromatic aldehyde in absolute ethanol gave the Schiff's bases (C3-C6). Treating a phenylacetic acid in triethylamine with Schiff's bases (C3-C6) in the presence of SOCl₂ to form the appropriate β -lactams (C8-C10). Reaction of compound (C1) with glycine in sodium hydroxide solution gave acid (C11). Oxazole derivatives was synthesized by the reaction of (C11) with different aromatic aldehyde in acetic acid and acetic

anhydride gave (C12,C13). Imidazole derivatives was synthesized by the reaction of hydrazine hydrate in dry benzene with different oxazole derivatives to give (C14,C15).Compounds (C14,C15) undergoes the character condensation reaction with different aromatic aldehyde in absolute ethanol gave Schiff's bases (C16-C19). Reaction of imine derivatives (C16-C19) with phenylacetic acid in the presence of SOCl₂ to form the another appropriate β -lactams (C20-C23). The product compounds was characterized by FT-IR, U.V and ¹HNMR spectrum.

KEYWORD: cinnamic acid, β -lactams, oxazole, Imidazole.

INTRODUCTION

In recent years, heterocyclic compounds had been receiving considerable attention due to their pharmacological and pesticidal importance.^[1-9] The simplest of five -membered

heterocyclic compounds are pyrrole, furan and thiophene, each of which contains a single hetero atom.^[10] Benzothiophene^[11] a class of heterocyclic compounds containing a benzene ring fused with five membered aromatic ring made up of one sulfur as heteroatom with formula C₈H₆S. Benzothiophene undergoes electrophilic aromatic substitution at C-2 and C-3 equally.^[12]

EXPERIMENTAL

Material and Instrument

IR spectra were recorded on (FTIR-8400s Fourier Transform infrared Spectrophotometer Shimadzu). UV Spectra were recorded on UV-Visible spectrophotometer (VARIAN) UV-1650PC. ¹H-NMR spectra (Burker DMX -500 NMR spectrophotometer) were recorded on ultra shield 300 MHz in Jordan, with tetramethylsilane as internal standard and DMSO as a solvent. Melting points were determined in a (Gallenkamp MFB-600- Melting point apparatus) melting point apparatus with sample contained in open capillary glass tube in an electrically heated metal block apparatus, and were uncorrected. All chemicals were supplied from BDH, Merck, Fluka and used without further purification.

Synthesis of 3-Chloro-1-benzothiophene-2-carbonyl chloride(C1)

Cinnamic acid (29.63 g, 0.20 mol), chlorobenzene (150 ml), and SOCl₂ (119 g, 1 mol) was stirred at room temperature for 30 min. Then, pyridine (1.58 g, 0.02 mol) was carefully added and the reaction mixture was refluxed for 72 hrs. The resulting residue was suspended in hot cyclohexane (400 ml) and immediately filtered. Upon staying at room temperature overnight, the title compound (C1) crystallized as yellowish needles.^[13]

Synthesis of 3-chlorobenzo[b]thiophene-2-carbohydrazide(C2)

A mixture of compound [C1] (2.29 g, 0.01 mol) and hydrazine hydrate (99%, 0.32 g, 0.01 mole) in benzene (25 mL) was refluxed for 4 hrs. Upon cooling the solution a solid appeared. This was recrystallized from ethanol to afford the desired compound(C2).^[14]

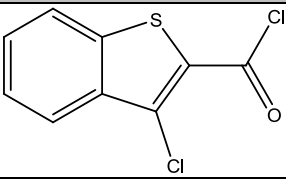
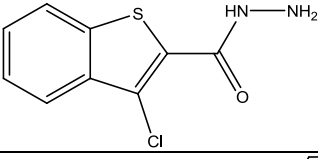
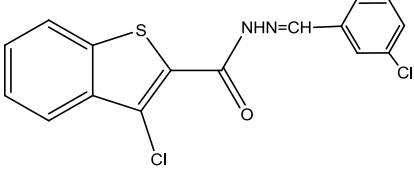
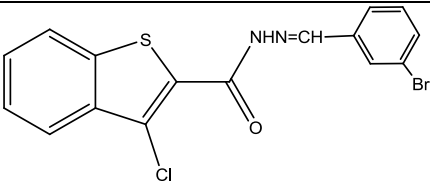
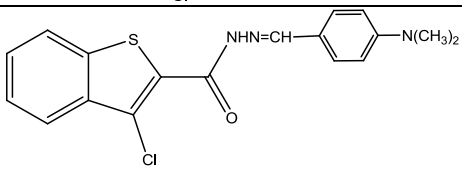
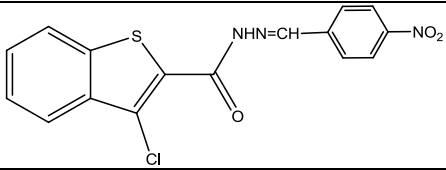
Synthesis of 3-chloro-N'-(Arylidene)benzo[b]thiophene-2-carbohydrazide(C3-C6)

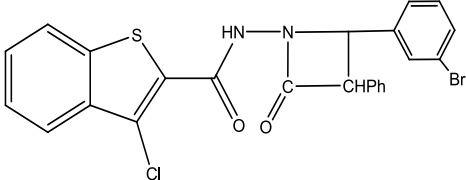
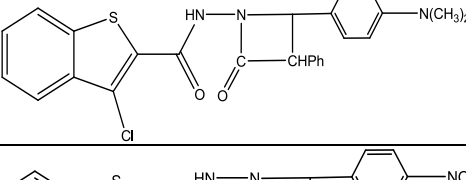
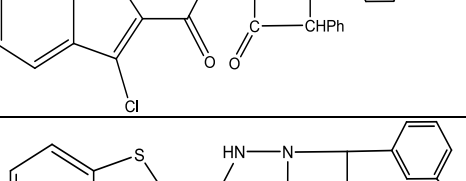
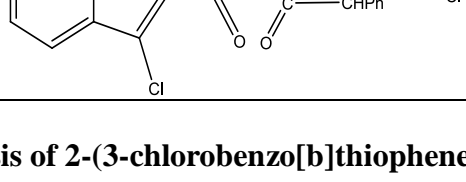
The corresponding aryl aldehyde (0.01 mol) was added to a stirred solution of compound (C2) (2.27 g, 0.01 mol) in absolute ethanol (30 ml) and the mixture was refluxed for 8 hrs. After cooling, the mixture was filtered and the solid recrystallized from ethanol to afford the desired compound(C3-C6).^[15]

Synthesis of N-(2-(Aryl)-4-oxo-3-phenylazetidin-1-yl)-3-chlorobenzo[b]thiophene-2-carboxamide(C7-C10)

To a mixture of phenylacetic acid (1.35 g , 0.01mol), imine (C3-C6) (0.01mol) and triethylamine (4.04 g , 0.04mol) in dry dichloromethane (40 ml) at 0°C, a solution of SOCl₂ (15 ml) in dry dichloromethane(20 ml) was added as drop wise . The mixture was stirred 24 hrs at room temperature. Then the mixture were washed successively with 1N HCl (30ml),water (3×30ml), and 5% NaHCO₃ (30ml). The organic layer was separated and dried over anhydrous sodium sulphate (Na₂SO₄), and then filtered solvent was removed under reduce pressure .The solid recrystallized from DMSO:H₂O to afford the desired compound. The physical properties are listed in table(1).^[16]

Table (1): Physical properties of compounds [C1 – C10]

Comp. No	Compound structure	Colour	Yield%	M.P	Purification solvent
C1		yellow	73	111-113	
C2		orange	92	128-130	ethanol
C3		Dark yellow	69	117-119	ethanol
C4		Light yellow	64	122-124	ethanol
C5		maroon	72	138-140	ethanol
C6		Deep yellow	86	143-145	ethanol

C7		Wight yellow	83	156-158	DMSO:H ₂ O
C8		Deep red	69	167-169	DMSO:H ₂ O
C9		orange	92	174-176	DMSO:H ₂ O
C10		olive	71	161-163	DMSO:H ₂ O

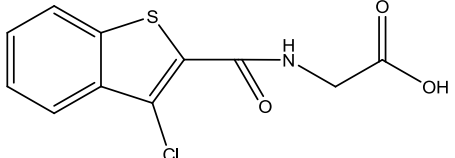
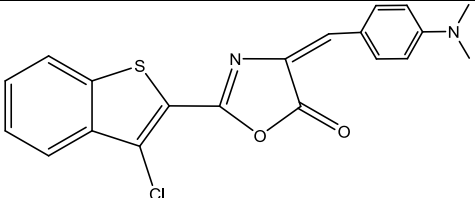
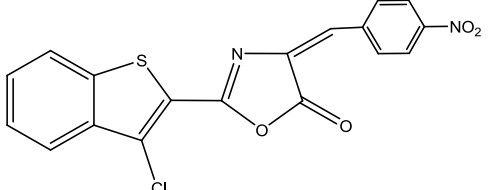
Synthesis of 2-(3-chlorobenzo[b]thiophen-2-carboxamido)acetic acid(C11)

Compound (C1) (2.29 g, 0.01 mol) was added to a stirring solution of glycine (0.75 g, 0.01 mol) and sodium hydroxide (50 ml, 10% solution). Then, the reaction mixture was shaken vigorously for 1 hr, and a few grams of crushed ice were added with stirring. After that, the solution was acidified with conc. HCl and the product was collected and recrystallized from ethanol.^[17]

Synthesis of 2-(3-chlorobenzo[b]thiophen-2-yl)-4-(Arylidene)oxazol-5(4H)-one (C12,C13)

Aromatic aldehyde (0.01 mol) was added to a stirring mixture of compound(C11) (2.69 g, 0.01 mol) acetic acid (5 ml) and acetic anhydride (20 ml). The temperature of reaction was increased to 70 °C for 10 min., then the mixture was poured into crushed ice and stirred for 30 min. the product was collected and recrystallized from ethanol to afforded the desired compound. The physical properties are listed in table(2).^[18]

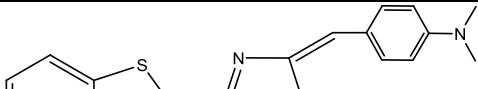
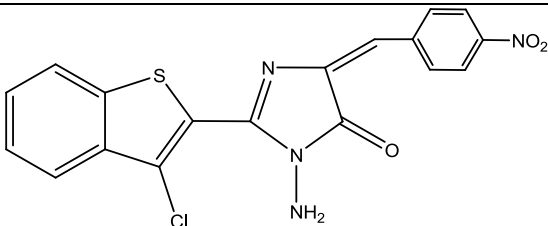
Table (2): Physical properties of compounds (C11 – C13)

Comp. No	Compound structure	Colour	Yield %	M.P	Purification solvent
C11		brown	89	124-126	ethanol
C12		Dark red	93	137-139	ethanol
C13		Bright orange	84	143-145	ethanol

Synthesis of 1-amino-2-(3-chlorobenzo[b]thiophen-2-yl)-4-(Arylidene)-1H-imidazol-5(4H)-one(C14,C15)

Hydrazine hydrate (99%, 10 ml) was added to a mixture of compound (C12,C13) (0.01 mol) in dry benzene (5 ml). The reaction mixture was refluxed for 20 hrs after cooling the solid product was obtained the desired compound. The physical properties are listed in table(3).^[19]

Table (3): Physical properties of compounds (C14 , C15]

Comp. No	Compound structure	Colour	Yield %	M.P	Purification solvent
C14		Deep red	86	138-140	ethanol
C15		Deep orange	92	144-146	ethanol

Synthesis of 2-(3-chlorobenzo[b]thiophen-2-yl)-4-(Arylidene)-1-(Aryl)-1H-imidazol-5(4H)-one(C16-C19)

The corresponding aryl aldehyde (0.01 mol) was added to a stirred solution of compound (C14-C15) (0.01 mol) in absolute ethanol (30 ml) and the mixture was refluxed for 8 hrs. After cooling, the mixture was filtered and the solid recrystallized from ethanol to afford the desired compound. The physical properties are listed in table(4).

Table (4): Physical properties of compounds (C16 – C19)

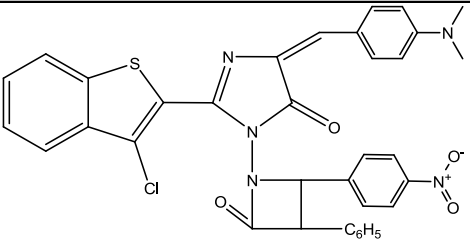
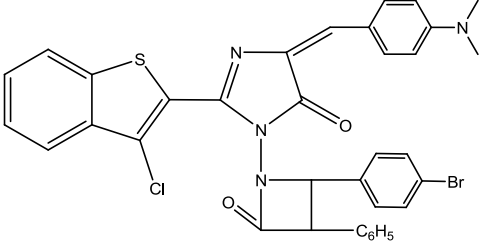
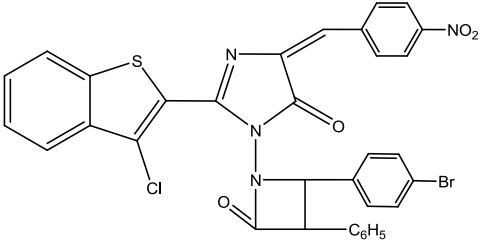
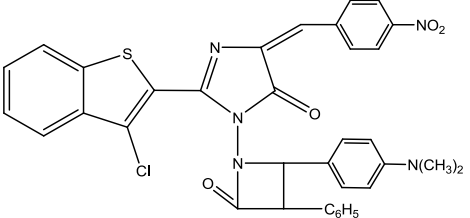
Comp. No	Compound structure	Colour	Yield %	M.P	Purification solvent
C16		maroon	82	161-163	ethanol
C17		red	80	173-175	ethanol
C18		yellow	86	168-170	ethanol
C19		orange	85	178-180	ethanol

Synthesis of 2-(3-chlorobenzo[b]thiophen-2-yl)-4-(Arylidene)-1-(2-(Aryl)-4-oxo-3-phenylazetidin-1-yl)-1H-imidazol-5(4H)-one(C20-C23)

To a mixture of phenylacetic acid (1.35 g , 0.01mol), imine (C16-C19) (0.01mol) and triethylamine (4.12 g , 0.04mol) in dry dichloromethane (40 ml) at 0°C, a solution of SOCl₂ (15 ml) in dry dichloromethane(20 ml) was added as drop wise . The mixture was stirred 24 hrs at room temperature. Then the mixture were washed successively with 1N HCl

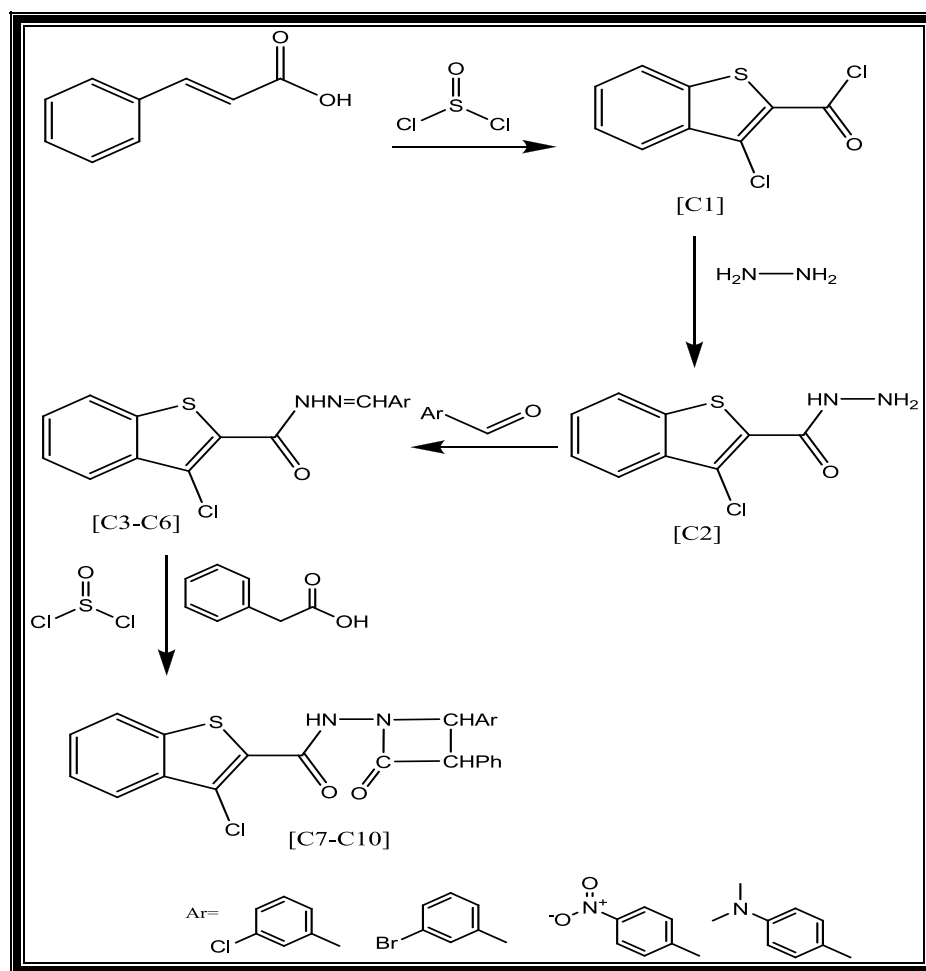
(30ml), water (3×30ml), and 5% NaHCO₃ (30ml). The organic layer was separated and dried over anhydrous sodium sulphate (Na₂SO₄), and then filtered solvent was removed under reduce pressure. The solid recrystallized from DMSO:H₂O to afford the desired compound. The physical properties are listed in table(5).

Table (5): Physical properties of compounds (C20 – C23)

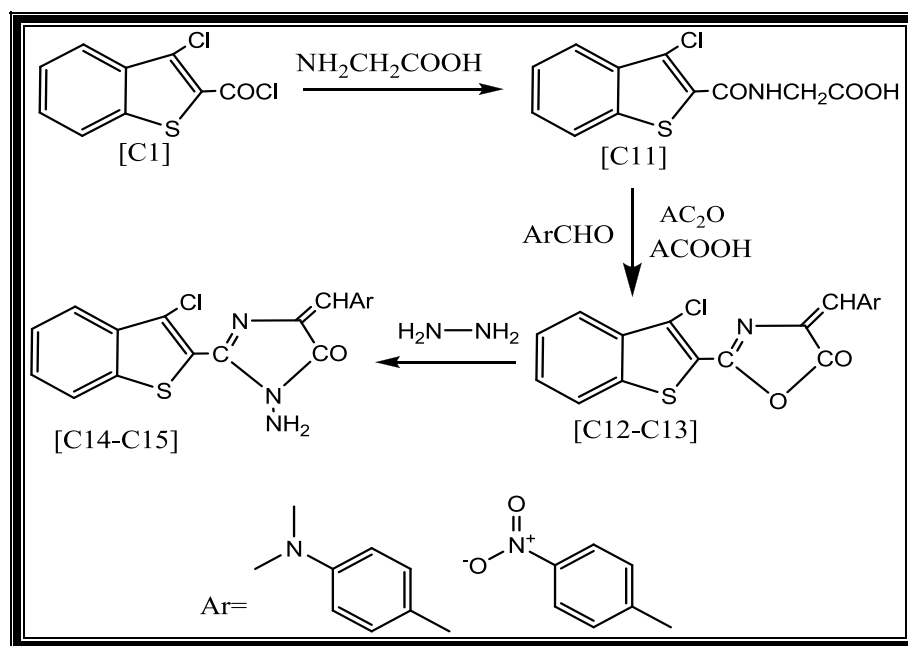
Comp. No	Compound structure	Colour	Yield %	M.P	Purification solvent
C20		orange	80	194-196	DMSO:H ₂ O
C21		red	85	185-187	DMSO:H ₂ O
C22		olive	77	177-179	DMSO:H ₂ O
C23		golden	76	173-175	DMSO:H ₂ O

RESULT AND DISCUSSION

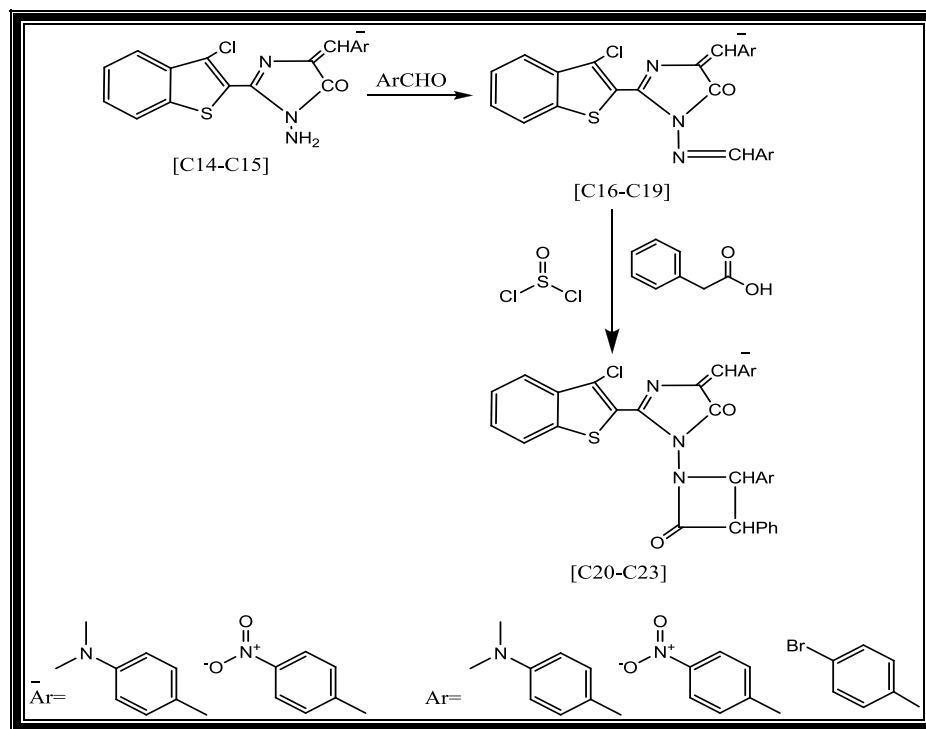
For the synthesis of the targeted compounds (C1- C27), the reaction sequences are outlined in scheme (1-3) from cinnamic acid.



Scheme (1)



Scheme (2)



Scheme (3)

Synthesis 3-Chlorobenzo[b]thiophene-2-carbonyl chloride(C1)

For synthesis of the target compounds (C1) which was prepared by the reaction of cinnamic acid and thionyl chloride. The structure of compound was confirmed by FT-IR and U.V spectrum. The FT-IR spectrum of compound (C1) (Fig. 1), table (6) shows disappearing of stretching vibration of (OH) group of carboxylic acid at $(2400-3400) \text{ cm}^{-1}$ and increasing frequency of (C=O) to $(1763) \text{ cm}^{-1}$, also spectrum shows another bands, $(3059) \text{ cm}^{-1}$ for aromatic (C-H) and band at $(1597-1479) \text{ cm}^{-1}$ due to aromatic (C=C). U.V. spectrum showed two absorption λ_{max} at 232 nm ($\pi-\pi^*$) and 344nm ($n-\pi^*$) electronic transition.

Synthesis of 3-chlorobenzo[b]thiophene-2-carbohydrazide(C2)

This compound was obtained by the reaction of 3-Chlorobenzo[b]thiophene-2-carbonyl chloride(C1) with hydrazine hydrate. The structure of compound (C2) was confirmed by FT-IR, U.V and ¹HNMR spectrum. FT-IR spectrum of compound (C2), table (6) shows the following bands, two bands at $(3317-3286) \text{ cm}^{-1}$ due to stretching vibration (asymmetric and symmetric) for (NH₂) group, band at $(3146) \text{ cm}^{-1}$ due to stretching vibration of (NH), decrease stretching vibration of carbonyl group to $(1653) \text{ cm}^{-1}$. Spectrum also shows other characteristic bands in table (6). UV spectrum of compound (C2) shows intense maxima at (268 nm) and (328 nm) due to $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ electronic transition, respectively.

The $^1\text{H-NMR}$ of compound (C2), table(6) shows the following signals.

- Singlet at (12.40) ppm due to (NH) group.
- Multiplate at (7.60-8.10) ppm due to aromatic proton
- Singlet at (4.72) ppm due to (NH_2) group.

Synthesis of 3-chloro-N'-(Arylidene)benzo[b]thiophene-2-carbohydrazide(C3-C6)

Compound (C2) undergoes the character condensation reaction with different aromatic aldehyde in absolute ethanol to give the Schiff's bases. The formation of these Schiff's bases was indicated by the disappearance of the NH_2 stretching vibration band. The structure of compound (C5) was confirmed by FT-IR and U.V spectrum. FT-IR spectrum of compound (C5), table (6) shows the following bands, band at $(3300)\text{ cm}^{-1}$ due to stretching vibration of (NH) group, and band at $(1615)\text{ cm}^{-1}$ for stretching vibration of (C=N) group. Spectrum also shows other characteristic bands in table (6). UV spectrum of compound (C5) shows intense maxima at (248 nm) and (339 nm) due to $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ electronic transition, respectively.

Table (6): IR and $^1\text{H-NMR}$ spectral data for compounds (C1 – C6)

Comp. No	Characteristic IR bands Cm^{-1}						$\text{H}^1\text{-NMR}$ ppm (DMSO\ 300 MHz)
	NH	CH aromatic	C=O	C=N	C=C aromatic	other	
C1		3059	1763		1597-1452		
C2	3146	3053	1653		1597-1479	3317-3286 (NH_2)	s(12.4)(NH), s(4.72) (NH_2), m(7.60-8.10) aromatic proton
C3	3334	3053	1660	1618	1589-1516	3167(N=CH), 752 (C-Cl)	
C4	3215	3051	1643	1620	1589-1546	3191(N=CH), 828(C-Br)	
C5	3300	3051	1656	1615	1589-1514	3120(N=CH), 2933-2868 (CH_3)	
C6	3201	3050	1654	1610	1600-1550	3120(N=CH), 1521-1365 (NO_2)	

Synthesis of N-(2-(Aryl)-4-oxo-3-phenylazetidin-1-yl)-3-chlorobenzo[b]thiophene-2-carboxamide(C7-C10)

Synthesis of β -lactams were done by treating a phenylacetic acid with triethylamine to form anion which then producing cycloaddition reaction with imine in the presence of SOCl_2 to form the appropriate β -lactams (C7-C10). The formation of these β -lactams was indicated by

the appearance of the two stretching vibration bands ($1722-1662\text{ cm}^{-1}$) to carbonyl group. The structure (C7) was confirmed by FT-IR, U.V and $^1\text{H-NMR}$ spectrum. FT-IR spectra, table (7) shows the stretching vibration bands at (3167 cm^{-1}) due to (NH) group, band at (3057 cm^{-1}) due to aromatic (C-H), band at ($2978-2901\text{ cm}^{-1}$) aliphatic (CH), band at ($1726-1664\text{ cm}^{-1}$) for (C=O) group and band at ($1575-1514\text{ cm}^{-1}$) for aromatic (C=C). Spectrum also shows other characteristic bands in table (7). UV spectrum of compound (C7) shows intense maxima at (285 nm) and (348 nm) due to $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ electronic transition, respectively.

The $^1\text{H-NMR}$ of compound (C7), table(7) shows the following signals:

- Singlet at (12.6) ppm due to (NH) group.
- Doublet at (7.94-7.95) ppm due to aromatic proton near $\text{N}(\text{CH}_3)_2$ group.
- Doublet at (7.61-7.62) ppm due to proton of aromatic ring of benzothiophen.
- multiplet at (7.51-7.54) ppm due to proton of aromatic ring.
- Doublet at (6.99-7.21) ppm due to aromatic proton far $\text{N}(\text{CH}_3)_2$ group.
- Doublet at (5.23-5.25) ppm due to (CHCO) group.
- Doublet at (4.60-4.66) ppm due to (CHCN) group.
- Singlet at (2.50) ppm due to (CH_3) group.

Table (7): IR and $^1\text{H-NMR}$ spectral data for compounds (C7 – C10)

Comp. No	Characteristic IR bands Cm^{-1}						$^1\text{H-NMR}$ \ ppm (DMSO \ 300 MHz)
	NH	CH aromatic	CH aliphatic	C=O	C=C aromatic	other	
C7	3167	3057	2978-2901-2847	1726-1664	1575-1514		s(12.6)(NH), d(7.94-7.95) near $\text{N}(\text{CH}_3)_2$, d(7.61-7.62) aromatic ring of benzothiophen, m(7.51-7.54) aromatic ring, d(6.99-7.21) far $\text{N}(\text{CH}_3)_2$, d(5.23-5.25) (CHCO) group, d(4.60-4.66) (CHCN) group. s(2.50) to (CH_3) group.
C8	3225	3061	2980-2899	1720-1662	1595-1523	1586-1346 (NO_2)	
C9	3194	3061	2928-2850	1720-1689	1577-1516	756 (C-Cl)	
C10	3201	3061	2982-2890	1722-1701	1591-1512	835 (C-Br)	

Synthesis of 2-(3-chlorobenzo[b]thiophene-2-carboxamido)acetic acid(C11)

This compound was synthesized by the reaction of compound (C1) with glycine in sodium hydroxide solution. The formation of (C11) was indicated by the appearance of the two stretching vibration bands to carbonyl group and the appearance of the two stretching vibration bands to NH, OH group. The structure (C11) was confirmed by FT-IR, U.V and ^1H NMR spectrum. FT-IR spectrum, table (8) shows the stretching vibration bands at (3390) cm^{-1} due to (NH) group, (3200-2400) cm^{-1} broad band for (OH) group, band at (3061) cm^{-1} due to aromatic (C-H), band at (2983-2939) cm^{-1} due to aliphatic (CH), band at (1726-1685) cm^{-1} due to (C=O) and band at (1597-1533) cm^{-1} due to for aromatic (C=C). UV spectrum of compound (C11) shows intense maxima at (257nm) and (317 nm) due to $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ electronic transition, respectively.

The ^1H -NMR of compound (C11), table(8) shows the following signals.

- Singlet at (12.70) ppm due to (OH) group.
- Singlet at (9.65) ppm due to (NH) group.
- Multiblet at (7.47-8.11) ppm due to aromatic proton.
- Singlet at (4.11) ppm due to (CH_2) proton.

Synthesis of 2-(3-chlorobenzo[b]thiophen-2-yl)-4-(Arylidene)oxazol-5(4H)-one (C12,C13)

This title compounds were synthesized according to the reaction of acid(C11) with different aromatic aldehyde in acetic acid and acetic anhydride to give (C12-C14). The formation of title compounds were indicated by the disappearance of the two stretching vibration bands to NH, OH groups and appearance of the one stretching vibration bands to carbonyl group (1759-1811) cm^{-1} . The structure (C12) was confirmed by FT-IR, U.V and ^1H NMR spectrum. FT-IR spectrum, table (8) shows the stretching vibration bands at (3057) cm^{-1} due to (=CH) group, band at (3014) cm^{-1} due to aromatic (C-H), band at (2922-2862-2816) cm^{-1} due to aliphatic (CH), band at (1761) cm^{-1} due to (C=O), band at (1669) cm^{-1} due to (C=N) and band at (1597-1527) cm^{-1} due to aromatic (C=C). Spectrum also shows other characteristic bands in table (8). UV spectrum of compound (C12) shows intense maxima at (298 nm) and (322 nm) due to $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ electronic transition, respectively.

The ^1H -NMR of compound (C12), table(8) shows the following signals.

- doublet at (7.83-7.88) ppm due to aromatic proton far $\text{N}(\text{CH}_3)_2$.
- doublet at (7.80--7.82) ppm due to aromatic proton near $\text{N}(\text{CH}_3)_2$.

- multiblet at(7.52-7.68) ppm due to aromatic proton for benzo[b]thiophen.
- Singlet at(6.24) ppm due to (CH) proton.
- Singlet at (2.55) ppm due to (CH₃) proton.

Table (8): IR and ¹H-NMR spectral data for compounds (C11 – C13)

Comp. No	Characteristic IR bands Cm ⁻¹						H ¹ -NMR\ ppm (DMSO\ 300 MHz)
	=CH	CH aromatic	C=O	C=N	C=C aromatic	other	
C11		3061	1726 acid, 1685 amide		1597-1533	3390(NH),3200-2400(OH),2983-2939 aliphatic (CH)	s(12.70)(OH) , s(9.65)(NH), m(7.47-8.11) aromatic proton , s(4 .11) aliphatic(CH)
C12	3057	3014	1786	1639	1597-1427	2922-2862 aliphatic (CH)	d(7.83-7.88) far N(CH ₃) ₂ , d (7.80-7.82) near N(CH ₃) ₂ ,m(7.52-7.68) aromatic benzo[b]thiophene ,s(6.24) for (=CH),s(2.55) aliphatic (CH ₃)
C13	3109	3055	1795	1656	1595-1552	1514-1340 (NO ₂)	

Synthesis of 1-amino-2-(3-chlorobenzo[b]thiophen-2-yl)-4-(Arylidene)-1H-imidazol-5(4H)-one(C14,C15)

This compounds were synthesized when Hydrazine hydrate in dry benzene was refluxed with different oxazole to give (C14-C15). Title compound was indicated by the appearance of the stretching vibration bands(3348-3271) cm⁻¹ to NH₂ groups and decreasing of stretching vibration bands (1664-1641) cm⁻¹ to carbonyl groups .The product(C15) was confirmed by FT-IR and U.V. spectrum. FT-IR spectrum, table (9) shows the stretching vibration bands at(3317-3288) cm⁻¹ due to stretching vibration (asymmetric and symmetric)for (NH₂) group ,band at (3090) cm⁻¹ due to for (=CH) group ,band at (3050) cm⁻¹ due to aromatic (C-H), band at (1653) cm⁻¹ due to(C=O) group, band at(1629) cm⁻¹ due to (C=N) group and band at (1599-1481) cm⁻¹ due to for aromatic (C=C) . Spectrum also shows other characteristic bands in table (9). UV spectrum of compound (C15) shows intense maxima at (281 nm) and (322 nm) due to $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ electronic transition, respectively.

The ¹H-NMR of compound (C15), table(9) shows the following signals.

- Doublet at (8.18-8.21) ppm due to aromatic proton far (NO₂).
- Doublet at (7.82-7.83) ppm due to aromatic proton near (NO₂).

- Multiblet at (7.57-7.60) ppm due to aromatic proton for benzo[b]thiophene.
- Signals at (6.41) ppm due to (=CH) proton.
- Signals at (4.70) ppm due to (NH₂) proton.

Table (9): IR and ¹H-NMR spectral data for compounds (C14 – C15)

Comp. No	Characteristic IR bands Cm ⁻¹							H ¹ -NMR\ ppm (DMSO\ 300 MHz)
	NH ₂	=CH	CH aromatic	C=O	C=N	C=C aromatic	other	
C14	3348-3329	3217	3030	1664	1627	1599-1525	2980-2887 aliphatic (CH)	
C15	3317-3288	3090	3050	1653	1629	1599-1481	1519-1309 for(NO ₂)	d(8.18-8.21)far NO ₂ , d(7.82-7.83) near NO ₂ m(7.57-7.60) aromatic benzo[b]th-iophene, s(6.41) for (=CH), s(4.70) for(NH ₂)

Synthesis of 2-(3-chlorobenzo[b]thiophen-2-yl)-4-(Arylidene)-1-(Aryl)-1H-imidazol-5(4H)-one(C16-C19)

Compound (C14-C15) undergoes the character condensation reaction with different aromatic aldehyde in absolute ethanol to give the Schiff bases (C16-C19) . The formation of these Schiff's bases was indicated by the disappearance of the NH₂ stretching vibration bands. The product(C17) was confirmed by FT-IR and U.V spectrum. FT-IR spectrum, table (10) showed the stretching vibration bands at(3203)cm⁻¹ due to (N=CH) group, band at(3082) cm⁻¹ due to(=CH), band at(3047) cm⁻¹due to aromatic (C-H), band at(2941-2808) cm⁻¹due to aliphatic (CH),band at (1651) cm⁻¹due to (C=O),band at(1626-1608)cm⁻¹ due to(C=N) , band at(1587-1523) cm⁻¹ due to for aromatic (C=C) and band at (858) cm⁻¹ due to for(Br) group . Spectrum also shows other characteristic bands in table (10). UV spectrum of compound (C17) shows intense maxima at (252 nm) and (338 nm) due to $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ electronic transition, respectively.

Table (10): IR and UV spectral data for compounds (C16 – C19)

Comp. No	U.V\ λ_{max} nm (DMSO)	Characteristic IR bands Cm^{-1}						
		N=CH	=CH	CH aromatic	C=O	C=N	C=C aromatic	other
C16	249 335	3219	3082	3043	1662	1643-1627	1599-1485	(2980-2862) aliphatic (CH), (1519-1340) (NO_2)
C17	252 338	3203	3082	3047	1651	1626-1608	1587-1523	(2941-2808) aliphatic (CH), (858) (C-Br)
C18	266 327	3181	3072	3023	1653	1608-1602	1550-1502	(1523-1365) (NO_2), (813) (C-Br)
C19	273 331	3165	3090	3053	1660	1618-1604	1582-1492	(2949-2860) aliphatic (CH), (1512-1380) (NO_2)

Synthesis of 2-(3-chlorobenzo[b]thiophen-2-yl)-4-(Arylidene)-1-(2-(Aryl)-4-oxo-3-phenylazetidin-1-yl)-1H-imidazol-5(4H)-one(C20-C23)

Synthesized of β -lactams were done by treating a phenylacetic acid with triethylamine to form anion which then producing cycloaddition reaction with imine in the presence of SOCl_2 to form the appropriate β -lactams (C20-C23). The formation of these β -lactams was indicated by the appearance of the two carbonyl stretching vibration bands ($1681\text{--}1781\text{ cm}^{-1}$). The product (C21) was confirmed by FT-IR and U.V spectrum. FT-IR spectra, table (11) shows the stretching vibration bands at (3109 cm^{-1}) due to (=CH), band at (3059 cm^{-1}) due to aromatic (C-H), band at ($2962\text{--}2924\text{ cm}^{-1}$) due to aliphatic (CH), band at ($1781\text{--}1681\text{ cm}^{-1}$) due to (C=O), band at (1634 cm^{-1}) due to (C=N), band at ($1593\text{--}1539\text{ cm}^{-1}$) due to for aromatic (C=C) and band at (866 cm^{-1}) due to for (Br) group. Spectrum also shows other characteristic bands in table (11). UV spectrum of compound (C21) shows intense maxima at (253 nm) and (378 nm) due to $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ electronic transition, respectively.

The $^1\text{H-NMR}$ of compound (C22), table (11) shows the following signals.

- Doublet at (8.27-8.28) ppm due to aromatic proton far (NO_2).
- Doublet at (8.19-8.24) ppm due to aromatic proton near (NO_2).
- Doublet at (8.17-8.18) ppm due to aromatic proton near (Br).
- Doublet at (8.02-8.15) ppm due to aromatic proton far (Br).
- Multiblet at (7.61-7.95) ppm due to aromatic proton for benzo[b]thiophene.
- Multiblet at (7.48-7.61) ppm due to for aromatic ring proton.

- Signals at (6.34) ppm due to (=CH) proton.
- Doublet at (4.82-4.84) ppm due to (NCH) of β -lactams ring.
- Doublet at (4.32-4.34) ppm due to (CHCO) proton.

Table (11): IR , UV, and ^1H -NMR spectral data for compounds (C20 – C23)

Comp . No	Characteristic IR bands Cm^{-1}							$\text{H}^1\text{-NMR}\backslash$ ppm (DMSO\ 300 MHz)
	=CH	CH arom	CH aliph	C=O	C=N	C=C arom	other	
C20	3059	3030	2976-2929	1747-1728	1658	1597-1575	(1514-1329) for (NO_2)	
C21	3109	3059	2962-2924	1781-1681	1634	1593-1539	(866) for (C-Br)	
C22	3061	3032	2994-2856	1734-1687	1646	1593-1505	(1516-1313) for (NO_2)	d (8.27-8.28) far (NO_2), d(8.19-8.24) near (NO_2), d (8.17-8.18) near (Br), d (8.02-8.15) far (Br), m(7.61-7.95) for benzo[b]thiophene, m(7.48-7.61) aromatic ring ,s (6.34)for (=CH) , d (4.82-4.84) (NCH) of B-lactam ring, d (4.32-4.34) (CHCO) proton.
C23	3061	3020	2978-2929	1722-1697	1632	1587-1492	(1514-1313) for (NO_2), (831) for (C-Br)	

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