

RESEARCH ARTICLE



**UTILIZING OF (Z)-5-AMINO-6,11-DIOXO-2-(PARA SUBSTITUTED PHENYLIMINO)-
1,2,3,6,11,11A-HEXAHYDROBENZO[G]IMIDAZO[1,2-A]QUINOLINE-4-CARBONITRILE FOR
SYNTHESIS OF NEW SPIRO AZITENONE AND THIAZOLIDINONE DERIVATIVES**

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Abstract

The new Schiff's bases was synthesised by the reaction of 5-amino-2,6,11-trioxo-1,2,3,6,11,11a-hexahydrobenzo[g]imidazo[1,2-a]quinoline-4-carbonitrile (**2**) with various aromatic amine and various nitroso compound to afford the corresponding (3a-c) and (6a-d), these compounds undergo cyclo addition reaction to afford the corresponding azetinone (**4a-c**), (**7a-d**) and (**5a-c**), (**8a-d**).

Keywords: Azetinone, thiazolidinone, Schiff's bases, spiro system, synthesised.

Introduction

-lactam have been reported to show biologically activities and also as antibiotics (Randhawa and Jamwal, 2011). Also -lactam antimicrobial agents (Alcaide and Almedros, 2001; Katzung, 1998), new class of -lactum antibiotics which have single monocyclic structure, Also called monobactams, were discovered and some of them have nature origin also isolated (Rode and Dobrowolski, 2003), Also synthetic -lactum use as antibacterial agents (Bose et al., 2000).

-lactum derivatives have been reported to show antimalarial activity (Nivsarkar et al., 2005), anti-Bacillus agent (Turos et al., 2006), anti-MRSA (Heldreth et al., 2006), Antitubercular activity (Pushkal et al., 2011), Anti-inflammatory activity (Khokra et al., 2011)] and anticancer activity (Banik et al., 2004), anti-oxidant activity (Mohammad et al., 2010). Recently discovered

antitumor monocyclic and bicyclic -lactum system (Giacomini and Cainelli, 2005), -lactum compound are considered bioactive molecule (. Shestakova and Vorona, 2004).

Schiff base possess antitubercular and anticancer activity (Pawar and Vibhute, 1999), compounds which contain quinoline nuclei have been shown to possess significant pharmacological activity such as anti-inflammatory, antifungal, antidepressant and anti-HIV infection (Dabholkar and Sager, 2007).

Chloroquinoline has been evaluated for their blood schizontocidal activity and reversal of chloroquine resistance activity (Tripathi and Saxena, 1995), antimalarial and antiviral activity (Biot and Daher, 2006), Azo dyes was found that to use in many organic synthesis (Zollinger, 1991).

Experimental details

All melting points are uncorrected, the reactions were monitored and the purity of products was controlled by Thin Layer Chromatography (TLC) using silica gel aluminium sheets 60F₂₅₄ (Merck, Germany). The IR spectra were obtained from KBr disks using Perkin Elmer 1650 ET-IR Spectrophotometer (USA). ¹H NMR spectra were recorded on Bruker AMX-250 spectrometer (Germany) at 250 MHz Mass spectra were recorded on Hewlett Packard Ms 5988 Spectrometer (USA). Elemental microanalyses were carried out on CE 440 Elemental Analyzer-Automatic Injector (Exeter Analytical, Inc., USA) at Cairo University, Cairo, Egypt.

Synthesis of (Z)-5-amino-6,11-dioxo-2-(para substituted phenylimino)-1,2,3,6,11,11a-hexahydrobenzo [g] imidazo [1,2-a]quinoline-4-carbonitrile (3a-c)

A mixture of **1** (10 mmol) and aromatic amine (10 mmol) refluxed in ethanol (30 mL) for 10-12 h. in presence of piperidine (0.5 mL). The reaction mixture was filtered at hot, evaporated under reduced pressure, residue poured onto ice/water, the solid precipitate was filtered and crystallized from DMF.

Synthesis of spiro-lactams (4a-c)

A mixture of **3a-c** (10 mmol) and chloroacetyl chloride (1.13 g, 10 mmol) was refluxed for 15 h. in ethanol (30 mL) in presence of triethylamine (0.5 mL). The mixture was filtered at hot, evaporated under reduced pressure. Residue thus obtained poured on to ice/water, the solid precipitate was filtered and crystallized from DMF.

Synthesis of spirothiazolidinone (5a-c)

A mixture of Schiff's bases **3a-c** (10 mmol) and mercaptoacetic acid (0.92 g, 10 mmol) in benzene (50 mL) was refluxed for 5 days poured on to ice cold water. The mixture was then separated using separator funnel. The solid product was filtered and crystallized from DMF.

(Z)-5-amino-1-(arylsubstituted)imino)-2,6,11-trioxo-1,2,3,6,11,11a-hexahydrobenzo[g]imidazo[1,2-a]quinoline-4-carbonitrile (6a-d)

A solution of **1** (2.99g, 10 mmol) in ethanol (30 mL) was treated with different aromatic nitroso

compounds (10 mmol), in the presence of piperidine as basic catalyst and heated under reflux for 15-16 h, (TLC control), filtered hot. The solvent was then evaporated under reduced pressure, the residue poured on to ice/water, filtered and crystallized from DMF.

4'-amino-3-chloro-1-(1-hydroxynaphthalen-2-yl)-2',4,6',11'-tetraoxo-3',6',11',11a'-tetrahydro-2'H-spiro[azetidine-2,1'-benzo[g]imidazo[1,2-a]quinoline (7a-d)

A mixture of **6a-d** (10 mmol) and chloroacetyl chloride (1.13 g, 10 mmol) was refluxed for 15 h. in ethanol (30 mL) in presence of triethylamine (0.5 mL). The mixture was filtered at hot, evaporated under reduced pressure. The residue treated with ice/water, the solid product thus formed filtered and crystallized from DMF.

4-amino-2'-(2-hydroxynaphthalen-1-yl)-4',6,11-trioxo-3,6,11,11a-tetrahydro-2H-spiro[benzo[g]imidazo[1,2-a]quinoline-1,3'-isothiazolidine]-5-carbonitrile(8a-d)

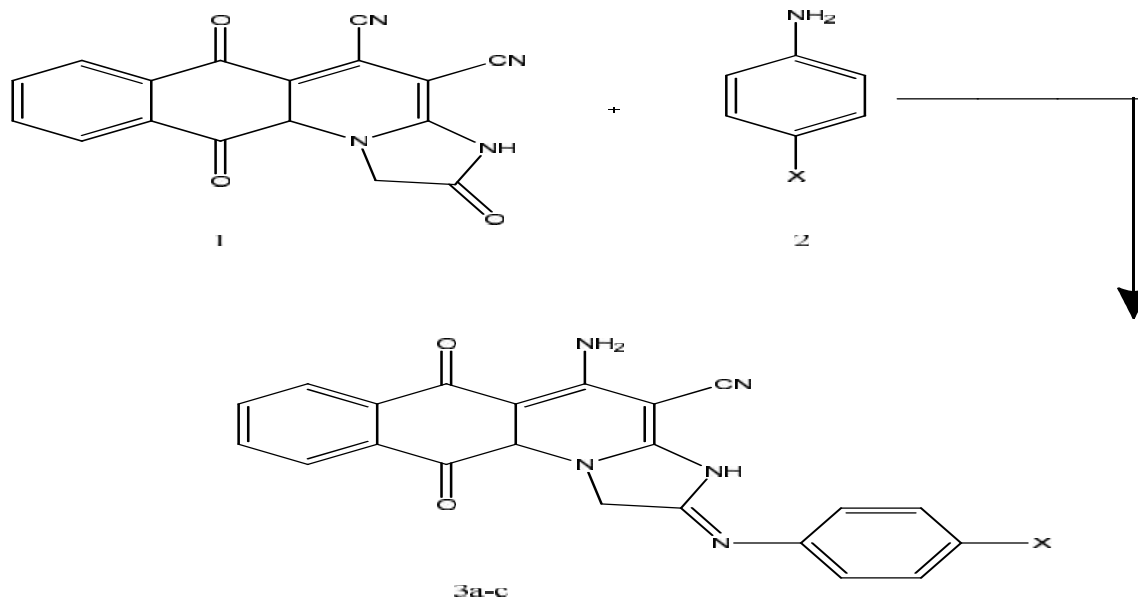
A mixture of **6a-d** (10 mmol) and mercaptoacetic acid (0.92 g, 10 mmol) in benzene (50 mL) was refluxed for 5 days poured on to ice cold water. The mixture was then separated using separator funnel. The solid product was filtered and crystallized from DMF.

Results and Discussion

Our approach to the synthesis of the desired spiro systems started with compounds **3a-c**. The activity of the carbonyl group in compound **1** (Elkanzi, 2014) prompted us to explore the possibility of utilizing some new Schiff bases prepared by condensation of **1** with an equimolecular ratio of aniline derivatives such as aniline and p-nitro aniline and p-hydroxy aniline in the presence of piperidine afford the corresponding new Schiff bases **3a-c**, the structure of these newly synthesised Schiff bases **3a-c** were confirmed by elemental analysis and IR, ¹HNMR and mass spectra [cf. Tables 1,2,3].

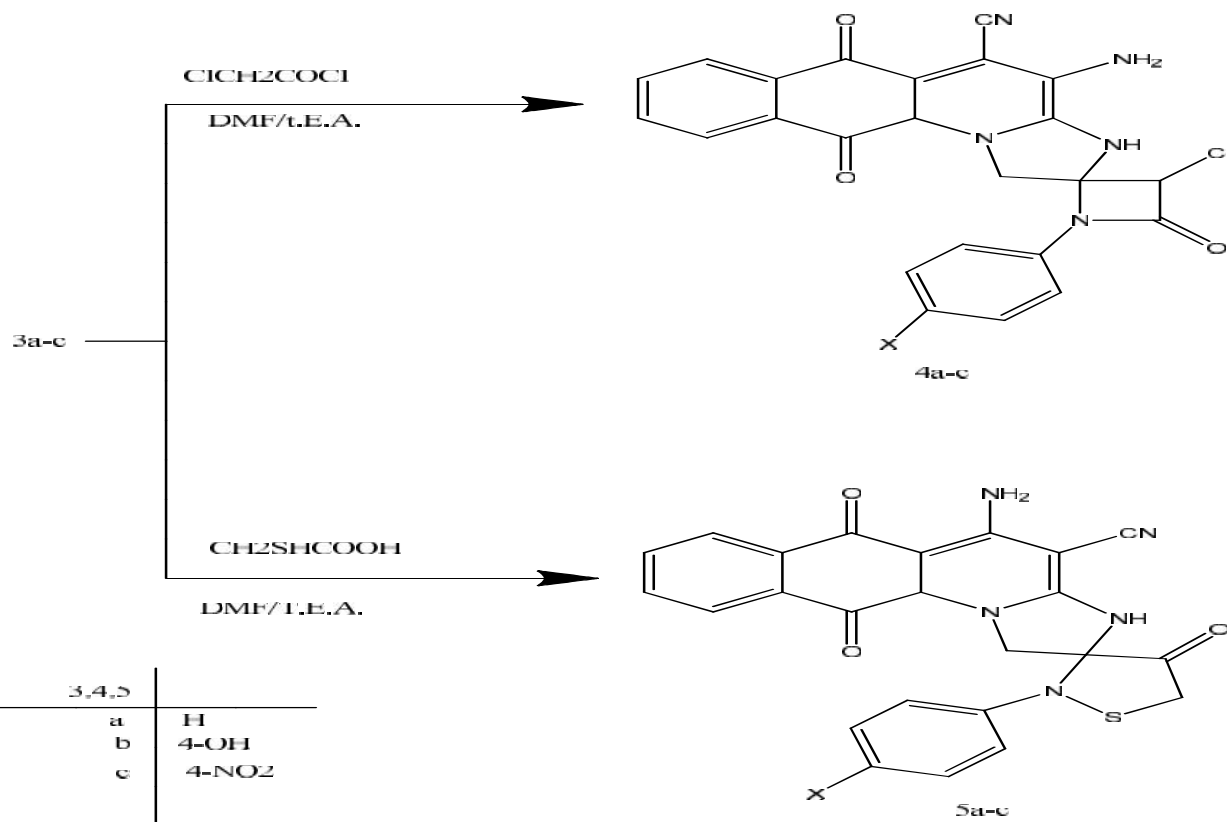
Our approach to the synthesis of the desired spiroβ-lactams containing a benzoquinolinoquinone nucleus started with the new Schiff's bases **3a-c** and equimolecular ratios of chloroacetyl chloride in dioxane in the presence of triethylamine catalyst (Khalafallah et al., 1993) to give **4a-c**. The structures of these compounds were confirmed by

Scheme 1



3	
a	H
b	4-OH
c	4-NO ₂

Scheme 2



3,4,5	
a	H
b	4-OH
c	4-NO ₂

Scheme 3

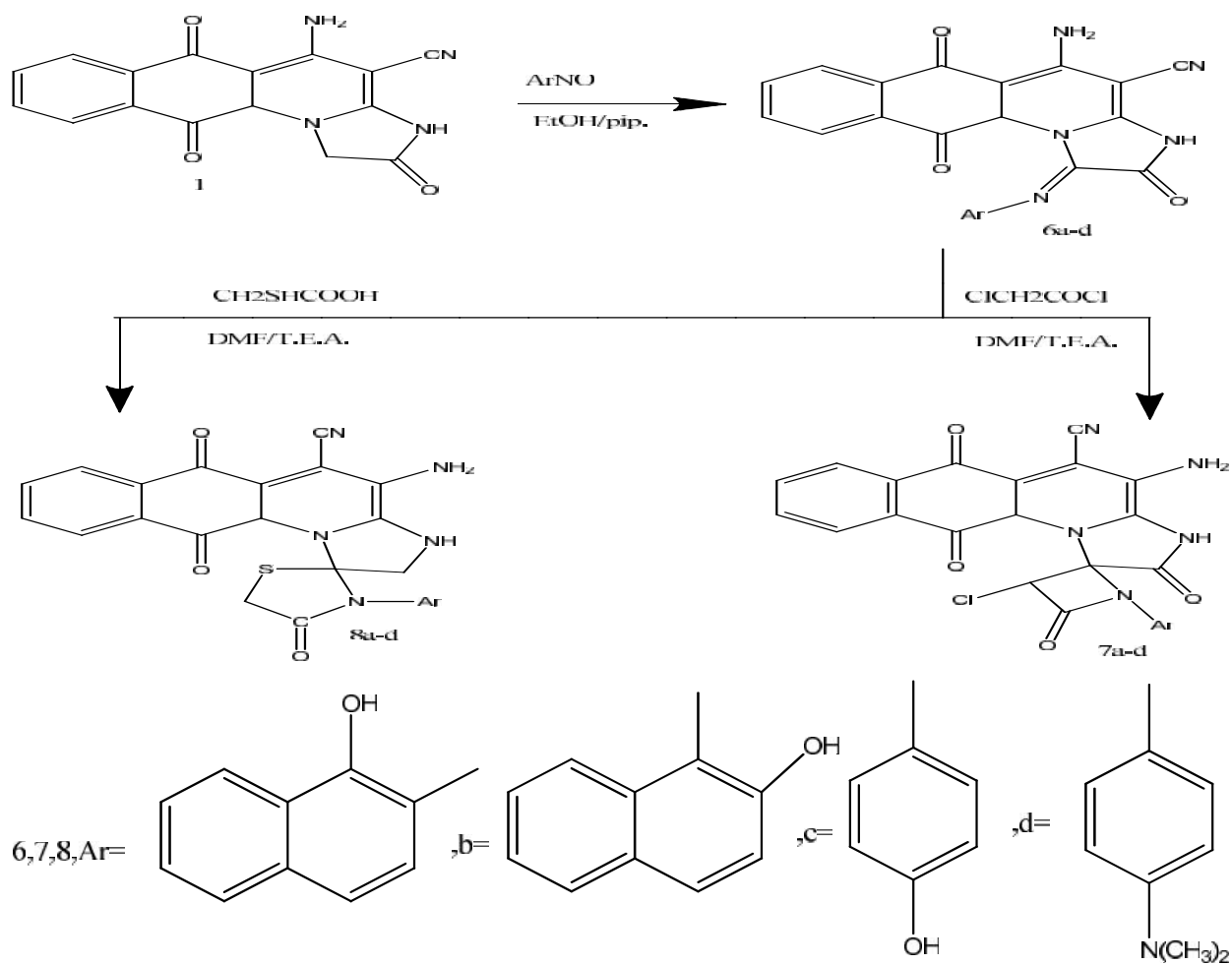


Table 1 Physical and Analytical Data of Compounds

Compound d	X	Ar	Time hours	MP (°C)	Yield %	Molecular Formula	Analysis % Calcd./Found				
							C	H	N	S	Cl
3a	H	-	10	>300[a]	67	C ₂₂ H ₁₅ O ₂ N ₅	69.28 69.43	3.96 3.68	18.36 18.39	- -	- -
3b	4-OH	-	11	>300[a]	65	C ₂₂ H ₁₅ O ₃ N ₅	66.49 66.64	3.80 3.52	17.62 16.63	- -	- -
3c	4-NO ₂	-	12	>300[a]	66	C ₂₂ H ₁₄ O ₂ N ₅ N ₆	61.97 60.84	3.31 2.93	19.71 19.01	- -	- -
4a	H	-	15	>300[a]	68	C ₂₄ H ₁₆ O ₃ N ₅ Cl	62.96 63.07	3.52 3.26	15.30 15.29	- -	7.74 7.73
4b	4-OH	-	15	>300[a]	69	C ₂₄ H ₁₆ O ₄ N ₅ Cl	60.83 60.93	3.40 3.16	14.78 14.78	- -	7.48 7.45
4c	4-NO ₂	-	15	>300[a]	67	C ₂₄ H ₁₅ O ₅ N ₆ Cl	57.32 56.64	3.01 2.70	16.71 16.19	- -	7.05 6.81
5a	H	-	120	>300[a]	73	C ₂₄ H ₁₇ O ₃ N ₅ S	63.29 63.40	3.76 3.53	15.38 15.38	7.04 7.02	- -

Table 1 Cont.
Physical and Analytical Data of Compounds

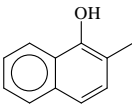
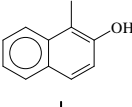
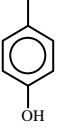
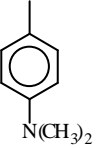
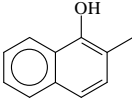
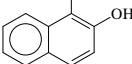
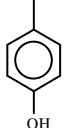
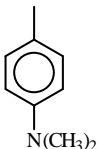
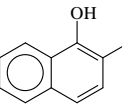
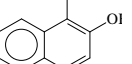
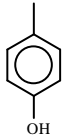
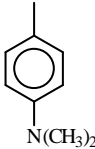
Compound	X	Ar	Time hours	MP (°C)	Yield %	Molecular Formula	Analysis % Calcd./Found				
							C	H	N	S	Cl
5b	4-OH	-	120	>300[a]	75	C ₂₄ H ₁₇ O ₄ N ₅ S	61.14 61.24	3.63 3.41	14.85 14.85	6.80 6.76	- -
5c	4-NO ₂	-	120	>300[a]	75	C ₂₄ H ₁₆ O ₅ N ₆ S	57.60 55.89	3.22 2.91	16.79 16.26	5.41 6.17	- -
6a	-		15	>300[a]	79	C ₂₆ H ₁₅ O ₄ N ₅	67.68 67.65	3.28 3.24	15.18 15.13	- -	- -
6b	-		15	>300[a]	73	C ₂₆ H ₁₅ O ₄ N ₅	67.68 67.64	3.28 3.25	15.18 15.14	- -	- -
6c	-		16	>300[a]	80	C ₂₂ H ₁₃ O ₄ N ₅	64.23 64.20	3.19 3.15	17.02 -	- -	- -
6d	-		16	>300[a]	83	C ₂₄ H ₁₈ O ₃ N ₆	65.75 65.71	4.14 4.10	19.17 19.15	- -	- -
7a	-		15	>300[a]	77	C ₂₈ H ₁₆ O ₅ N ₅ Cl	62.54 62.50	3.00 2.98	13.02 13.00	- -	6.59 6.55
7b	-		15	>300[a]	74	C ₂₈ H ₁₆ O ₅ N ₅ Cl	62.54 62.51	3.00 2.96	13.02 13.01	- -	6.59 6.53

Table 1 Cont.
Physical and Analytical Data of Compounds

Compound	X	Ar	Time hours	MP (°C)	Yield %	Molecular Formula	Analysis % Calcd./Found				
							C	H	N	S	Cl
7c	-		15	>300[a]	76	C ₂₄ H ₁₄ O ₅ N ₅ Cl	59.09 59.05	2.89 2.86	14.36 14.33	- -	7.27 7.26
7d	-		15	>300[a]	73	C ₂₆ H ₁₉ O ₄ N ₆ Cl	60.65 60.61	3.72 3.70	16.32 16.28	- -	6.89 6.85
8a	-		120	>300[a]	78	C ₂₈ H ₁₉ O ₄ N ₅ S	64.48 64.43	3.67 3.62	13.43 13.35	6.15 6.04	- -
8b	-		120	>300[a]	75	C ₂₈ H ₁₉ O ₄ N ₅ S	64.48 64.45	3.67 3.63	13.43 13.37	6.15 6.01	- -

8c	-		120	>300[a]	75	C ₂₄ H ₁₇ O ₄ N ₅ S	61.14 61.09	3.63 3.60	14.85 14.81	6.80 6.78	- -
8d	-		120	>300[a]	77	C ₂₆ H ₂₂ O ₃ N ₆ S	62.64 62.61	4.45 4.42	16.86 16.82	6.43 6.41	- -

[a] From DMF

Table 2 . elemental analysis, IR, ¹HNMR and mass spectra

Comp. No.	¹ H NMR (DMSO) μ ppm
3a	2.84(s, 2H), 6.73(s, NH ₂), 8.02-7.1(m, 10H), 10.26(s, NH).
3b	2.89(s, 2H), 6.75(s, NH ₂), 8.02-7.1(m, 9H), 9.35(brs, OH), 10.29(s, NH).
3c	2.86(s, 2H), 6.72(s, NH ₂), 8.02-7.1(m, 9H), 10.32(s, NH).
4a	2.91(s, 2H), 6.77(s, NH ₂), 8.02-7.1(m, 11H), 10.34(s, NH).
4b	2.93(s, 2H), 6.79(s, NH ₂), 8.02-7.1(m, 10H), 9.37(brs, OH), 10.42(s, NH).
4c	2.90(s, 2H), 6.75(s, NH ₂), 8.02-7.1(m, 10H), 10.46(s, NH).
5a	2.95(s, 2H), 2.52(s, 2H, CH ₂), 6.81 (s, NH ₂), 8.02-7.1(m, 10H), 10.65(s, NH).
5b	2.98(s, 2H), 2.51(s, 2H, CH ₂), 6.83 (s, NH ₂), 8.02-7.1(m, 9H), 9.42(brs, OH), 10.67(s, NH).
5c	2.99(s, 2H), 2.52(s, 2H, CH ₂), 6.78 (s, NH ₂), 8.02-7.1(m, 9H), 10.69(s, NH).
6a	6.77(s, NH ₂), 8.02-7.1(m, 11H), 9.46(brs, OH), 10.76(s, NH).
6b	6.78(s, NH ₂), 8.02-7.1(m, 11H), 9.44(brs, OH), 10.77(s, NH).
6c	6.75(s, NH), 8.02-7.1(m, 9H), 10.79(s, NH), 9.36(brs, OH).
6d	1.85(s, 6H), 6.56(s, NH ₂), 8.02-7.1(m, 9H), 10.75(s, NH).
7a	6.82(s, NH ₂), 8.02-7.1(m, 12H), 9.27(brs, OH), 10.97(s, NH).
7b	6.85(s, NH ₂), 8.02-7.1(m, 12H), 9.29(brs, OH), 10.99(s, NH).
7c	6.87(s, NH ₂), 8.02-7.1(m, 10H), 9.32(brs, OH), 10.96(s, NH).
7d	1.92(s, 6H), 6.89(s, NH ₂), 8.02-7.1(m, 10H), 10.95(s, NH).
8a	2.52(s, 2H, CH ₂), 6.85 (s, NH ₂), 8.02-7.1(m, 13H), 9.28(brs, OH), 11.21(s, NH).
8b	2.51(s, 2H, CH ₂), 6.87 (s, NH ₂), 8.02-7.1(m, 13H), 9.31(brs, OH), 11.23(s, NH).
8c	2.50(s, 2H, CH ₂), 6.82 (s, NH ₂), 8.02-7.1(m, 11H), 9.35(brs, OH), 11.25(s, NH).
8d	1.89(s, 6H), 2.50(s, 2H, CH ₂), 6.79 (s, NH ₂), 8.02-7.1(m, 11H), 11.22(s, NH).

their elemental analysis, IR, ¹HNMR and mass spectra [cf. Tables 1,2,3].

We were synthesized new spirothiazolidinones compounds **5a-c** by the cyclo addition of thioglycolic acid (1:1 molar ratio) to Schiff bases **3a-c** in boiling benzene using a water separator system for five days (Khalafallah et al., 1993) afforded the corresponding **5a-c**. The structures of compounds **5a-c** were confirmed by elemental analysis, IR, ¹HNMR and mass spectra [cf. Tables 1,2,3].

Our approach to the synthesis of the desired spiro systems started with compounds **6a-d**, which were prepared by condensation of nitroso compounds such as α -nitroso β -naphthol, β -nitroso- α -naphthol, p-nitroso-phenol and p-nitroso-N-dimethylaniline with compound **1** in absolute ethanol and in the

presence of piperidine catalyst. The structures of compounds **6a-d** were confirmed by elemental analysis, IR, ¹HNMR and mass spectra [cf. Tables 1,2,3].

Synthesis of spiro compounds was achieved through the interaction of **6a-d** with equimolar ratios of chloroacetyl chloride in dimethylformamide in the presence of triethylamine catalyst (Khalafallah et al., 2002) to give **7a-d**. The structures of these compounds were confirmed by elemental analysis, IR, ¹HNMR and mass spectra [cf. Tables 1,2,3].

Spiro thiazolidinone **8a-d** were prepared by the cycloaddition of thioglycolic acid (1:1 molar ratio) to the previously prepared Schiff bases **6a-d** in boiling benzene for five days (Khalafallah et al., 2002)

using a water separator system afford the corresponding compounds **8a-d**.

Table 3. Elemental analysis, IR, ¹HNMR and mass spectra

Comp. No.	IR $\nu_{\max}/\text{cm}^{-1}$	Mol.wt	MS
3a	3365(NH), 3210(NH ₂), 2225(C≡N), 1617(C=N), 1690(C=O).	381.39	380(M ⁺)
3b	3395(OH), 3380(NH), 3205(NH ₂), 2228(C≡N), 1619(C=N), 1685(C=O).	397.39	397
3c	3380(NH), 3213(NH ₂), 2226(C≡N), 1620(C=N), 1689(C=O).	442.38	442
4a	3379(NH), 3195(NH ₂), 2220(C≡N), 1684(CO), 1648(CO).	457.87	459(M ⁺²)
4b	3390(OH), 3365(NH), 3190(NH), 2223(C≡N), 1689(CO), 1650(CO).	473.87	474(M ⁺¹)
4c	3390(NH), 3175(NH ₂), 2219(C≡N), 1693(CO), 1653(CO).	502.87	502
5a	3375(NH), 3165(NH ₂), 2215(C≡N), 1695(CO), 1660(CO).	455.11	455
5b	3370(OH), 3365(NH), 3170(NH ₂), 2218(C≡N), 1690(CO), 1665(CO).	471.49	471
5c	3380(NH), 3185(NH ₂), 2216(C≡N), 1689(C=O), 1657(CO).	500.49	500
6a	3397(OH), 3375(NH), 3175(NH ₂), 2217(C=O), 1655(CO).	461.44	461
6b	3395(OH), 3370(NH), 3165(NH ₂), 2220(C≡N), 1618(C=N), 1695(C=O), 1650(CO).	461.44	462(M ⁺¹)
6c	3390(OH), 3360(NH), 3170(NH ₂), 2218(C≡N), 1622(C=N), 1689(C=O), 1645(CO).	411.38	412(M ⁺¹)
6d	3350(NH), 3210(NH ₂), 2223(C≡N), 1620(C=N), 1692(C=O), 1649(CO).	438.45	439(M ⁺¹)
7a	3395(OH), 3255(NH), 3205(NH ₂), 2225(C≡N), 1695(C=O), 1646(2CO).	537.93	537
7b	3387(OH), 3360(NH), 3210(NH ₂), 2227(C≡N), 1693(CO), 1650(2CO).	537.93	537
7c	3380(NH), 3360(NH), 3220(NH ₂), 2214(C≡N), 1680(C=O), 1658(2CO).	487.87	487
7d	3375(NH), 3215(NH ₂), 2216(C≡N), 1685(C=O), 1660(2CO).	514.93	515(M ⁺¹)
8a	3385(OH), 3360(NH), 3225(NH ₂), 2221(C≡N), 1693(C=O), 1665(2CO).	521.12	521
8b	3390(OH), 3365(NH ₂), 3319(NH ₂), 2223(C≡N), 1690(C=O), 1670(2CO).	521.12	521
8c	3380(OH), 3365(NH), 3216(NH ₂), 2225(C≡N), 1692(C=O), 1668(2CO).	471.49	471
8d	3389(OH), 3360(NH), 3195(NH ₂), 2226(C≡N), 1694(C=O), 1666(2CO).	498.15	498

The structures of compounds **8a-d** were confirmed by elemental analysis, IR, ¹HNMR and mass spectra [cf. Tables 1,2,3].

Conclusion

In this research work we synthesis new Schiff's bases from the reaction of compounds **1** With different aromatic amine or different aromatic aldehydes to afford the corresponding **3a-c** and **6a-d**, also these new Schiff's bases undergo

cyclocondensation reaction to give the corresponding spiro beta lactam and spirothiazolidinone **4a-a,7a-d** and **5a-c,8a-d**.

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