

**SYNTHESIS, CHARACTERIZATION AND ANTIMICROBIAL EVALUATION OF  
SCHIFF BASE 4-THIAZOLIDINONES OF AMINOSALICYLIC ACID DERIVATIVES**

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Article Received on 12/08/2022

Article Revised on 02/09/2022

Article Accepted on 22/09/2022

**ABSTRACT**

Keeping in view the pharmacological potential of thiazolidinones and 4-aminosalicylic acid derivatives, the title compounds containing these nuclei were synthesized. Reaction of 4-aminosalicylic acid (I) with substituted 2-chloro(3a-d), 2-hydroxy(4a-d) and 2-thio(5a)-3-formylquinolines (M1-9) in presence of catalytic amount of glacial acetic acid in refluxing ethanol afford Schiff's bases. Further, cyclization of compounds (M1-9) with thioglycolic acid in 1, 4-dioxan in presence of anhydrous  $ZnCl_2$  furnished desired novel compounds (MS1-9). The newly synthesized compounds were characterized by spectroscopic and physical methods. All the synthesized compounds were screened for antibacterial and antifungal activity by standard methods. Results of the anti-microbial study reveals that, some compounds exhibited moderate to good anti-bacterial and anti-fungal activities.

**KEYWORDS:-** p-aminosalicylic acid, thiazolidinone, Quinoline, Cyclocondensation, vilsmeier reagent, Schiff's bases, thioglycolic acid.

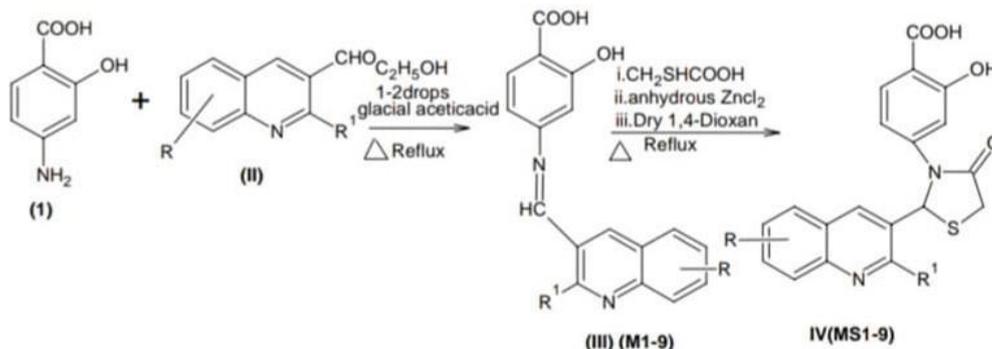
**INTRODUCTION**

The development of anti-microbial drugs represent one of the most important advance in therapeutic for cure and control of the serious infections or in prevention and treatment of infectious complication of other therapeutic modalities as cancer chemotherapy and surgery. Development of resistance to current anti-bacterial therapy continues to stimulate the search for more effective agents. In addition, primary and opportunistic fungal infections continue to increase rapidly because of the increased number of immuno compromised patients (AIDS, Cancer, and transplants). Several reviews have appeared illustrating the problems encountered by increasing contemporary clinicians dealing with infectious diseases. Hence in the present work we have attempted to explore the possibility of getting novel antimicrobial agents by linking 4-thiazolidinone of amino salicylic acid containing substituted 2-chloro, 2-hydroxy and 2-thio-3-formylquinolines.<sup>[1-2]</sup> Thus obtained 4-thiazolidinone of amino salicylic acid derivatives, will be screened for the anti-bacterial and anti-fungal activities. For the proposed work 4-aminosalicylic acid nucleus was selected as a synthon. This was linked with various substituted quinoline aldehydes to form the corresponding Schiff base intermediates.<sup>[3]</sup> These intermediates were converted into final thiazolidinones analogues by using thioglycolic acid

in 1, 4-dioxan used as a solvent in presence of anhydrous zinc chloride. Thus newly synthesized compounds will be characterized by analytical and spectral (IR,  $^1H$ -NMR and Mass) properties.<sup>[4]</sup> All the compounds will be screened for anti-bacterial and anti-fungal activities by MIC method.

**MATERIALS AND METHODS**

As the aim of the present work is to synthesize novel heterocyclic compounds containing 4-thiazolidinone and quinoline nucleus, p-aminosalicylic acid which amino group at Para position is selected as synthon. Among the various types of quinoline molecules which contain various functionalities at various positions, substituted 2-Chloro, 2-hydroxy and 2-thio-3-formyl quinolines are selected to linking to p-aminosalicylic acid. This is because of fact that, amino group and aldehydes group containing compounds can be easily linked which will lead to formation of Schiff's bases.<sup>[5-6]</sup> Cyclocondensation of schiffs bases with thioglycolic acid in presence of dry 1, 4-dioxan and catalytic amount of  $ZnCl_2$  afforded of title compounds<sup>[7-8]</sup> is depicted below (**scheme-1**).



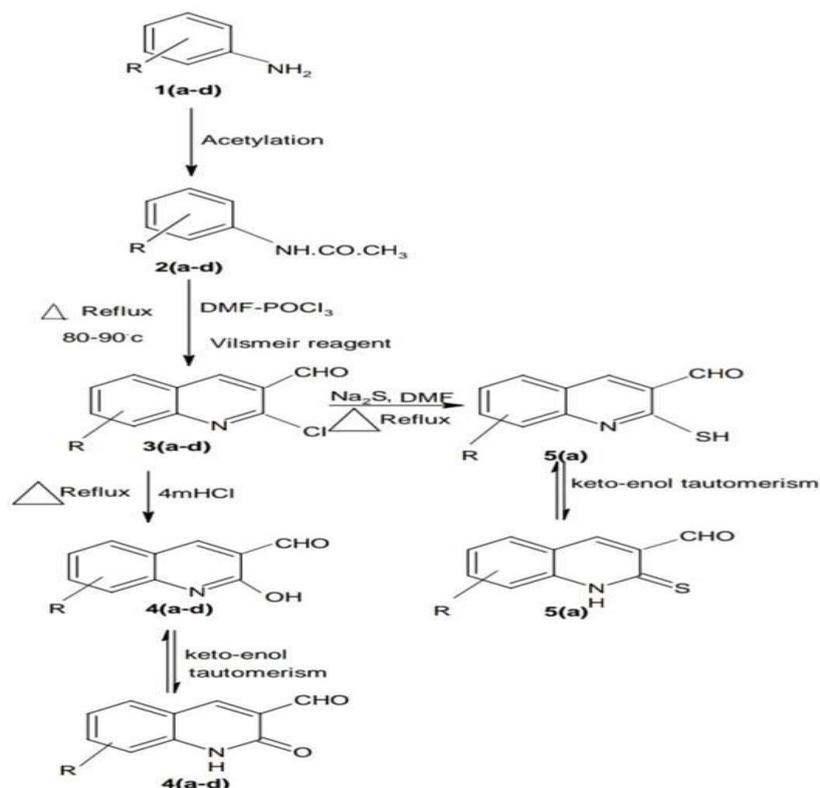
Scheme I

Compounds	R	R	R	R	R <sup>1</sup>
MS(1-4)	H	6-CH <sub>3</sub>	8-CH <sub>3</sub>	7-CH <sub>3</sub>	Cl
MS(5-8)	H	6-CH <sub>3</sub>	8-CH <sub>3</sub>	7-CH <sub>3</sub>	OH
MS(9)	H	-	-	-	SH

The required starting compound P-aminosalicylic acid obtained from Otto chemika- aiochemika reagents for synthesis in Ar grade. The authenticity of the compound will be confirmed by comparing the melting point of the compound with the literature melting point 145 °C 1-11.

Similarly, the required substituted 2-Chloro, 2-hydroxy

and 2-thione-3-formyl quinolines are prepared by adopting standard procedure by vilsmeier approach, involving the reaction of N- arylacetamides with vilsmeier reagent.<sup>[9-10]</sup> The products obtained in good yield and characterized by their physical constant and elemental analysis<sup>12-14, 21.</sup>



Compounds	R	R	R	R
1(a-d) and 2(a-d)	H	4-CH <sub>3</sub>	2-CH <sub>3</sub>	3-CH <sub>3</sub>
3(a-d) and 4(a-d)	H	6-CH <sub>3</sub>	8-CH <sub>3</sub>	7-CH <sub>3</sub>
5(a)	H	-	-	-

### Reaction of P-Aminosalicylic acid with different substituted 2-Chloro, 2-hydroxy and 2-thione-3-formyl quinolines:

The P-Aminosalicylic acid in ethanol has been condensed with nine substituted 2-Chloro, 2-hydroxy and 2-thione-3-formyl quinolines, in presence of catalytic amount of glacial acetic acid is expected to results in the formation of Schiff's bases. Various aromatic quinoline aldehydes will be selected based on rational bases that are aromatic quinoline aldehydes with electron withdrawing and electron donating groups will be selected. The resultant products have been purified by recrystallization in suitable solvent. The resultant products of Schiff's bases treated with thioglycolic acid in dry 1, 4-dioxan in presence of catalytic amount of ZnCl<sub>2</sub> by heating under reflux for 19-25 hrs.<sup>[11-12]</sup> TLC monitoring of the reaction has clearly indicated the formation of single spot. The resultant products have been purified and recrystallization from aqueous N,N-dimethylformamide and characterized as the respective 4-Thiazolidinones of Aminosalicylic acid derivatives **III (MS1-9)** yielded brown colour powder 274<sup>o</sup>C. For instance, 4-Amino salicylic acid (**1**) has been condensed by heating under reflux for 19-25 hrs with substituted 2-Chloro, 2-hydroxy and 2-thione-3-formyl quinolines in presence catalytic amount of glacial acetic acid in ethanol medium to yield the Schiff's bases (**M1-9**). Cyclocondensation of Schiff's bases with thioglycolic acid in presence of anhydrous ZnCl<sub>2</sub> in dry 1,4-dioxan medium refluxed for 19-25hrs. TLC monitoring of the reaction has clearly indicated the formation of single product. Afforded 2-Hydroxy-4-[4-oxo-2(quinolin-3yl)-1,3-thiazolidin-3yl]benzoic acid derivatives (MS1-9).

The product on purification by recrystallization from aqueous N, N-Dimethyl formamide has brown colour powders, M.P 274<sup>o</sup>C. (Table 1)

### Identification and Characterization

**Melting point determination:** The melting points of the organic compounds were determined by open capillary tube method. Melting point is a valuable criteria of purity for an organic compound as a pure crystal is having definite and sharp melting point. The purity should not be assumed but must be established by observation of any changes in the melting point when the compound is subjected to purification by recrystallization. The synthesized compounds showed a minute change in melting point after recrystallization.<sup>[13]</sup>

**Solubility:** The solubility of synthesized compounds was tested in various solvents.

The solubility characters were listed.

**Thin layer chromatography: Chromatography** is an important technique to identify the formation of

new compounds and also to determine the purity of the compound. The RF value is characteristic for each of the compound. In all the cases the distance traveled by the sample was found to be different from that of the parent compound spotted along with it. Thus confirming the fact that the compounds formed were entirely different from that of the parent compound. The R<sub>f</sub> value of compounds was reported in **Table-2**.

### Spectral studies<sup>[14-16]</sup>

**Infrared spectrum of the compound (MS-2):** (KBr Pellet method) showed

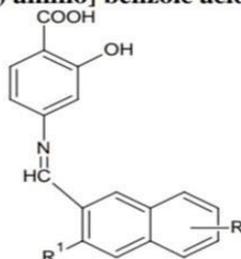
absorption bands at (in cm<sup>-1</sup>): 1714 (C=O), 1495 (C=C), 626 (C-Cl).

**<sup>1</sup>H-NMR spectrum of the compound (MS-2):** (in DMSO-d<sub>6</sub>) has been found to exhibit characteristic proton signals at (in ppm): 7.99 (s, 1H, -OH), 1.25 (s, 2H, -CH<sub>2</sub>), 3.30 (s, 1H, -CH), 2.15 (s, 3H, -CH<sub>3</sub>), 6.75-7.15 (m, 4H, Ar-H).

**Mass spectrum of the compound (MS-2):** The Mass spectrum of the compounds has recorded. Its molecular ion peak at m/z 414 (M<sup>+</sup>). On the basis of this spectral data, the compound could be characterized as the respective 4-[2-(2-chloro-7-methylquinolin-3-yl)-4-oxo-1,3-thiazolidin-3-yl]-2-hydroxybenzoic acid (MS-4) and 4-[2-(2-chloro-6-methylquinolin-3-yl)-4-oxo-1,3-thiazolidin-3-yl]-2-hydroxybenzoic acid (MS-2).

Similarly, P-Amino salicylic acid has been condensed as many as nine different substituted 2-Chloro, 2-hydroxy and 2-thione-3-formyl quinolines and the single product uniquely obtained on each such reaction could be characterized as their respective 2-Hydroxy-4-[4-oxo-2(quinolin-3yl)-1,3-thiazolidin-3yl]benzoic acid derivatives. Thus, nine of them have been prepared and their characterization data are presented in the **Table-1, 2 & 3**.

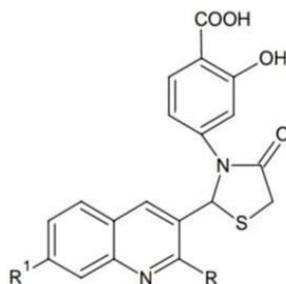
Table – 1  
Characteristic analytical data of 2-Hydroxy-4-[(quinolin-3yl-methylidene) amino] benzoic acid derivatives.



Sl.No	Comd. Code	M.P °C	% Yield	Mol. Formula	M.Wt.	Rf Value*	Calculated (%)		
							C	H	N
1	M-1	288	84	C <sub>17</sub> H <sub>11</sub> N <sub>2</sub> O <sub>3</sub> Cl	326.69	0.80	62.49	3.39	8.57
2	M-2	242	84	C <sub>18</sub> H <sub>13</sub> ClN <sub>2</sub> O <sub>3</sub>	340.72	0.64	63.44	3.85	8.22
3	M-3	230	80	C <sub>18</sub> H <sub>13</sub> N <sub>2</sub> O <sub>3</sub> Cl	340.72	0.95	63.44	3.85	8.22
4	M-4	246	78	C <sub>18</sub> H <sub>13</sub> N <sub>2</sub> O <sub>3</sub> Cl	340.72	0.76	63.44	3.85	8.22
5	M-5	256	81	C <sub>17</sub> H <sub>12</sub> N <sub>2</sub> O <sub>4</sub>	308.22	0.74	66.23	3.92	9.09
6	M-6	268	75	C <sub>18</sub> H <sub>14</sub> N <sub>2</sub> O <sub>4</sub>	322.24	0.68	67.07	4.38	8.69
7	M-7	280	79	C <sub>18</sub> H <sub>14</sub> N <sub>2</sub> O <sub>4</sub>	322.24	0.71	67.07	4.38	8.69
8	M-8	270	66	C <sub>18</sub> H <sub>14</sub> N <sub>2</sub> O <sub>4</sub>	322.24	0.57	67.07	4.38	8.69
9	M-9	290	71	C <sub>17</sub> H <sub>12</sub> N <sub>2</sub> O <sub>3</sub> S	326.32	0.83	62.95	3.73	8.64

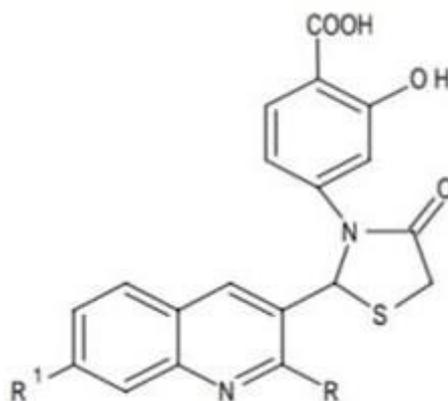
\*Solvent – Chloroform: alcohol, Ratio – 9: 1

Table – 2  
Characteristic analytical data of 2-Hydroxy-4-[4-oxo-2-(quinolin-3yl)1, 3-thiazolidin-3yl]benzoic acid derivatives.



Sl.No	Comp d. code	M.P °C	% Yield	Mol. Formula	M.Wt.	Rf Value *	Calculated (%)		
							C	H	N
1	MS-1	274	80	C <sub>19</sub> H <sub>13</sub> N <sub>2</sub> O <sub>4</sub> S	400.83	0.85	56.93	3.27	8.84
2	MS-2	294	80	C <sub>20</sub> H <sub>15</sub> N <sub>2</sub> O <sub>4</sub> S	414.86	0.78	57.90	3.64	6.75
3	MS-3	298	78	C <sub>20</sub> H <sub>15</sub> N <sub>2</sub> O <sub>4</sub> S	414.86	0.80	57.90	3.64	6.75
4	MS-4	287	88	C <sub>20</sub> H <sub>15</sub> N <sub>2</sub> O <sub>4</sub> S	414.86	0.82	57.90	3.64	6.75
5	MS-5	276	80	C <sub>19</sub> H <sub>14</sub> N <sub>2</sub> O <sub>5</sub> S	382.38	0.68	59.68	3.69	7.33
6	MS-6	268	66	C <sub>20</sub> H <sub>16</sub> N <sub>2</sub> O <sub>5</sub> S	396.41	0.76	60.60	4.07	7.07
7	MS-7	305	73	C <sub>20</sub> H <sub>16</sub> N <sub>2</sub> O <sub>5</sub> S	396.41	0.79	60.60	4.07	7.07
8	MS-8	299	76	C <sub>20</sub> H <sub>16</sub> N <sub>2</sub> O <sub>5</sub> S	396.41	0.68	60.60	4.07	7.07
9	MS-9	314	88	C <sub>19</sub> H <sub>14</sub> N <sub>2</sub> O <sub>4</sub> S <sub>2</sub>	398.45	0.73	57.27	3.54	7.03

\*Solvent – Chloroform: alcohol, Ratio – 9: 1

**Table 3: IR, NMR and Mass spectral characteristic analytical data of 2-hydroxy-4-[4-oxo-2(quinolin-3yl)-1, 3-thiazolidin-3yl] benzoic acid.**

Sl.No	Compd .code	C=O Cm <sup>-1</sup>	C = C Cm <sup>-1</sup>	C = Cl Cm <sup>-1</sup>	C - S Cm <sup>-1</sup>	<sup>1</sup> H NMR Spectral Values(δ values)	Mass spectral values (M <sup>+</sup> m/zvalues)
1	MS-1	1490	1716	-	-	8(s, 1H-OH), 1.25 (S, 2H, CH <sub>2</sub> ), 3.29(s, 1H, CH)	
2	MS-2	1495	1731	-	626	7.99(s, 1H-OH), 1.25 (S, 2H, CH <sub>2</sub> ), 3.30(s, 1H, CH),2.15(S,3H,CH <sub>3</sub> ),6.75-7.15(m,4H,Ar-H)	M/z 414(M <sup>+</sup> ), 414(M+1).
3	MS-3	1487	1716	-	764		
4	MS-4	1504	-	-	713	7.95(s, 1H-OH), 1.25 (S, 2H, CH <sub>2</sub> ), 3.71(s, 1H, CH),2.54(S,3H,CH <sub>3</sub> ),6.25-7.35(m,4H,Ar-H)	
5	MS-5	1497	-	-	755	7.88(s, 1H-OH), 1.25 (S, 2H, CH <sub>2</sub> ), 3.3(s, 1H, CH)	
6	MS-6	1435	1715	-	601		
7	MS-7	1506	-	-	772	7.94(s, 1H-OH), 1.24 (S, 2H, CH <sub>2</sub> ), 3.43(s, 1H, CH),2.57(S,3H,CH <sub>3</sub> ),6.44-6.95(m,4H,Ar-H)	
8	MS-8	1489	-	-	-		
9	MS-9	1489	-	611	-		

## RESULTS AND DISCUSSION

**Antibacterial activity<sup>[17]</sup>:** The antibacterial activity of the synthesized compounds MS1-9 was determined invitro using MIC (Broth Dilution Method) against four pathogenic microorganisms viz., *Escherichia coli*, *Pseudomonas aeruginosa* (Gm-ve) and *Staphylococcus aureus*, *Staphylococcus pyogenus* (Gm+ve) at various conc. Between 6.125 µg/ml to 1000 µg/ml. Out of test compounds MS- 9 exhibited high potent activity against *E. coli* and *S. pyogenus* at MIC of 12.5 µg/ml and 25 µg/ml respectively. MS-2, MS-5 and MS-8 showed equipotent activity against *E. coli*, MS-2, MS-5 and MS-9 showed equipotent activity against *P. aeruginosa*, MS1 -5 and MS-9 showed equipotent activity against *S. aureus*, MS-7 and MS-8 showed equipotent activity against *S. pyogenus*. Whereas rest of synthesized compounds exhibited feeble to moderate activity against all strains of tested

organisms when compared to reference standard ampicillin at 100 µg/ml.

The compound containing electron withdrawing group and unsubstituted phenyl ring favored more potent activity. (Table 4)

### Antifungal activity<sup>[18-20]</sup>

The antifungal activity of the synthesized compounds MS 1-9 was determined in-vitro using Agar plate method against three strains viz., *Candida albicans*, *Aspergillus niger* and *Aspergillus clavatus* at different conc. ranging between 100 µg/ml to 1250 µg/ml.

Out of the tested compounds MS-2, MS-3 and MS-4 exhibited a significant activity than that of standard at 500 µg/ml against *Candida albicans* at conc.

250µg/ml and 200 µg/ml respectively compared to reference standard Griseofulvin at conc. 500 µg/ml. MS-9 showed equipotent antifungal activity at (MIC 500µg/ml). Whereas rest of synthesized compounds

exhibited feeble to moderate activity against all strains of tested fungi when compared to reference standard Griseofulvin (MIC 500µg/ml).

**Table 4: Results of anti bacterial by mic method for the synthesized compounds (MIC IN µg/ml).**

S.No.	Compound Code	<i>Escherichiacoli</i> [MTCC 442] (Gm -ve)	<i>Pseudomona saeruginosa</i> [MTCC 441] (Gm -ve)	<i>Staphylococc us aureus</i> [MTCC 96] (Gm +ve)	<i>Staphylococcu spyogenus</i> [MTCC 443] (Gm +ve)
1	Ampicillin (Std)	100	100	250	100
2	MS -1	125	200	250	250
3	MS -2	100	100	250	250
4	MS-3	250	250	250	250
5	MS-4	250	200	250	250
6	MS -5	100	100	250	250
7	MS -6	125	125	100	125
8	MS -7	200	200	100	100
9	MS -8	100	125	200	100
10	MS -9	62.5	100	250	25

**Table 5: Results of antifungal activity by mic method for the synthesized compounds (MIC IN µg/ml).**

S.NO.	Compound code	<i>Candida albicans</i> [MTCC 227]	<i>Aspergillus niger</i> [MTCC 282]	<i>Aspergillus clavatus</i> [MTCC 1323]
1	Griseofulvin(std)	500	100	100
2	MS-1	1000	>1000	>1000
3	MS-2	250	>1000	>1000
4	MS-3	250	>1000	>1000
5	MS-4	250	500	500
6	MS-5	1000	250	250
7	MS-6	1000	250	250
8	MS-7	1000	500	500
9	MS-8	1000	250	250
10	MS-9	500	>1000	>1000

Std: Griseofulvin (Grisovin FP)

Mean minimum inhibitory concentration including conc. of drug in µg/ml.

Among the synthesized compounds, MS-9 exhibited high antibacterial activity against both Gm+ve and Gm-ve pathogens compared to other tested compounds. This compound contains a 2-sulfonyl group. This indicates a relationship between the structure and the anti-bacterial activity of the compound. Further research is required to get a clear idea about the relationship between anti-bacterial activity and structure of the compounds.

The results of antifungal activity summarized in Table-5 reveals that compounds MS-2, MS-3 and MS-4 exhibited a significant activity against tested fungal strains than any other tested compounds. These compounds contain 2-chloro groups respectively. However, further studies are required to come to a conclusion about establishing a correlation between antifungal activity and structure of the synthesized

compounds.

## CONCLUSION

Nine new compounds of 2-Hydroxy-4-[4-oxo-2(quinolin-3yl)-1, 3- thiazolidin- 3yl] benzoic acid derivatives were synthesized. Analytical and spectral data were used to characterize few synthesized compounds. All synthesized compounds were screened for antibacterial and antifungal activities. Tested compounds exhibited high potent to moderate antibacterial activity against *E. coli* and *S. pyogenus* (Gm-ve) organisms. Few of the tested compounds exhibited significant and equipotent antifungal activity against *Candida albicans*, but none of the synthesized compounds shown significant antifungal activity against *A.niger* and *A.clavatus*.

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