INTRODUCTION

4-Thiazolidinones play a vital role due to their wide range of biological activities and industrial importance. 4-Thiazolidinones are always being an attraction point for researchers because of its efficiency towards various pharmacological usages. The enhanced prevalence of infectious diseases threatens world population. Although tuberculosis appeared as a curable disease for years, it is regaining importance due to the multidrug resistance\(^1\);\(^2\). Worldwide statistics on tuberculosis surprisingly reveals that, nearly one–third of the world’s population is infected with tuberculosis, with approximately eight million new patients every year. A major issue is the increase of multi-drug resistant tuberculosis (MDRTB) giving rise to the disease expensive and incurable especially in immunod efficient subjects such as AIDS patients. Hence, there is an increased demand to develop new antituberculosis agents effective against pathogens resistant to current treatment.

The derivatives of 4-thiazolidinone nucleus have occupied a unique place in the field of medicinal chemistry due to wide range of biological activities like antibacterial, antitubercular, anticancer, anticonvulsant and antifungal\(^3\). They have interesting activity profiles mainly cox-1 inhibitors, inhibitors of bacterial enzyme, non nucleoside inhibitors of HIVRT and antihistaminic agents\(^4\). 4-thiazolidinones are derivatives of thiazolidinone with carbonyl group at the 4\(^{th}\) position and formed by the attack of sulphur nucleophile on imine carbon followed by intramolecular cyclisation with elimination of water.

Recent studies on molecular modification of the latter (TBZs) revealed that, dismantling of the imidazole nucleus leading to the design of new 1,3-thiazolidin-4-one derivatives, maintained the molecular requirements for enzyme inhibition\(^5\). A literature search revealed that, 4–thiazolidinone derivatives may exhibit antibacterial\(^6\);\(^7\), antituberculosis\(^8\)–\(^10\), antiviral\(^12\)–\(^15\),\(^11\)–\(^16\) and anticancer\(^17\);\(^19\) properties. According to Andres et al.\(^6\), 4-thiazolidinones may be considered as phosphate bioisosteres and therefore inhibit the bacterial enzyme MurB which is involved in the biosynthesis of peptidoglycan layer of the cell wall\(^6\). In addition, some thiazolidinones were recently reported as novel inhibitors of mycobacterial rhamnose synthetic enzymes\(^21\). This new approach is believed to be selective as rhamnose which is not found in humans, has been shown to be essential for mycobacterial cell wall synthesis\(^21\).
SYNTHETIC ASPECTS

Synthesis of 4-thiazolidinones has been reported either by cyclisation of acyclic compounds or by interconversion among appropriately substituted thiazolidinone derivatives.

1. R. A. Mane and co-workers have synthesised 4-thiazolidinone bearing 2-mercapto-4-methylimidazoles moiety.

2. Mogilaiah et al. have synthesized 2-aryl-3-(2-trifluromethyl-1,8-naphthyridine-3-carbonylamino)-4-thiazolidinones.

3. Synthesis of 4-thiazolidinones has been reported by the microwave irradiation.

4. Parikh et al. have synthesised variety of 4-thiazolidinones bearing diphenyl sulphone, substituted aryl arsanilic acid, 2-aryl-1,3,4-thiadiazole, γ-picolinylamino, s-triazine, benzoylmino acetamido, sulphonamido benzoylamin, phthalazine-1-yl-amino, aryl substituted hydroxyaryl, β-β-dichloroethylaminophenyl and 8-hydroxyquinolinyl moieties of 4-thiazolidinone ring system (V) have been reported as potent antimicrobial agents.

MECHANISM

The cyclocondensation proceeds by the attack of the mercaptoacetic acid upon the C=N group. The HSCH\(_2\)COOH adding to the carbon atom followed by the cyclisation.

Sometimes, the uncyclised product in these reaction has been isolated. Phosphorous pentoxide in dioxan was used for subsequent cyclisation of certain uncyclised products. The nucleophilic attack of thioglycolic anion on carbon of azomethine having positive character and nitrogen having negative character is...
Simultaneously, the removal of water takes place is rate determining step and helps in cyclisation.

**THERAPEUTIC EVALUATION**

The thiazolidinone derivatives substituted at 2- and 3-position are associated with diverse biological activities which have been reported as under.

1. Antidiabetic\(^{[39]}\)
2. Antiulcer\(^{[40]}\)
3. Insecticidal\(^{[41]}\)
4. Antiproteolytic\(^{[42]}\)
5. Antiparkinsonian\(^{[43]}\)
6. Thrombin Inhibitors\(^{[44]}\)
7. Antiinflammatory\(^{[45]}\)
8. Analgesic\(^{[46]}\)
9. Antifungal\(^{[47]}\)
10. Antimicrobial\(^{[48,49]}\)
11. CNS effect\(^{[50]}\)
12. Antihypertensive\(^{[51]}\)
13. Anticonvulsant\(^{[52]}\)
14. H1-Histamine antagonists\(^{[53]}\)

Tamura et al.\(^{[54]}\) have reported antimicrobial activity of 4-thiazolidinone derivatives. B. Lohary et al.\(^{[55]}\) have synthesised and reported hypolipidemic activity of 4-thiazolidinone derivatives. Bhawana et al.\(^{[56]}\) have assessed some new 4-thiazolidinones as antiinflammatory agents. Antifungal and antibacterial activity of thiazolidinones has been reported\(^{[57]}\). O. A. Fathalla\(^{[58]}\) have synthesised some new 4-thiazolidinones as anticancer and antibacterial agent. Mohie A. and co-workers\(^{[59]}\) investigated some new 4-thiazolidinones as anticancer agent. Antiinflammatory and analgesic activity of 4-thiazolidinones has been investigated\(^{[60]}\).

Siddique and co-workers\(^{[61]}\) have synthesised novel thiazolidinones and evaluated for the antithyroid activity. Mayer et al\(^{[62]}\) have prepared thiazolidinones and studied their herbicidal activity. Archana and Srivastava\(^{[63]}\) have synthesised new 4-thiazolidinones (II) as potent anticonvulsant agent. S. K. Srivastava et al\(^{[64]}\) have formulated some new 4-thiazolidinones (I) as antibacterial, antifungal, analgesic and diuretic agents.
Thiazolidinones useful in the treatment of inflammation have been synthesised\textsuperscript{[65]}, Govindarajan R. et al\textsuperscript{[66]} have described thiazolidinone derivatives of pyrazinoic acid and reported their antibacterial, antifungal and antitubercular activity. Thiazolidinones for the inhibition of phosphatases and the treatment of cancer have been reported\textsuperscript{[67]}.

Parikh et al. have synthesised variety of 4-thiazolidinones bearing diphenyl sulphone\textsuperscript{[68]}, substituted aryl\textsuperscript{[69,70]}, arsanilic acid\textsuperscript{[71]}, 2-aryl-1,3,4-thiadiazole\textsuperscript{[72]}, \(\gamma\)-picolinylamino\textsuperscript{[73]}, s-triazine\textsuperscript{[74]}, benzoylamino acetamido\textsuperscript{[75]}, sulphonamido benzoylamino\textsuperscript{[76]}, phthalazine-1-yl-amino\textsuperscript{[77]}, aryl substituted hydroxyaryl, \(\beta\)-\(\beta\)-dichloroethylaminophenyl and 8-hydroxyquinolinyl moieties of 4-thiazolidinone ring system have been reported as potent antimicrobial agents.

Moreover, H. H. Parekh and co-workers\textsuperscript{[78]} have synthesized new bis-4-thiazolidinones and studied their biological activity. A. R. Parikh and coworkers\textsuperscript{[79]} have assessed thiazolidinone derivatives bearing 7-methoxyquinoline nucleus for antimicrobial activity.

Dandia et al\textsuperscript{[80]} have synthesized thiazolidinone derivatives and reported their antifungal activity. Bioactive venlafaxine analogs such as 2,3-disubstituted-1,3-thiazolidinones have been synthesized and reported as antimicrobial agent by Kavitha et al.\textsuperscript{[81]} Denis et al.\textsuperscript{[82]} have synthesized 4-thiazolidinones derivatives by the cyclization unsymmetrical thioureas. Some nickel (II) complexes of 4-thiazolidinone has been synthesized by Dave et al.\textsuperscript{[83]} Various worker have applied the Green chemistry approach to the synthesis of 4-thiazolidinone derivatives by using microwave assisted method and multi component reaction method\textsuperscript{[84,85]}. 

\[
\text{(I)} \quad \text{(II)}
\]
Angela Rao et.al. have been synthesized some novel thiazolidinone derivatives (III, IV) and report anti HIV activity.

![Chemical Structure of III and IV](image)

K. H. Patel et. al. have synthesized 2-amino-6-(2-naphthalenyl) thiazolo [3,2-d]thiadiazole (V) by treatment of KCNS and Br₂ on 2-Amino-4-(2-naphthalenyl)thiazole. The prepared compounds have been screened on some stains of fungi.

![Chemical Structure of V](image)

Tejaskumar J. Shah et. al. have been described A series of 2-(substituted phenyl)-3-[4-(2,4-dichloro-5-fluorophenyl)-6-(2-thienyl)pyrimidine-2-yl-ureido]-5H/methyl/carboxymethyl-4-thiazolidinones(VI) and screened against different strains of bacteria and fungi.
Considerable evidence has been accumulated to demonstrate the wide applications of thiazolidinone derivatives and also Pyrazolo[3,4-b]pyridine nucleus have drawn the attention of chemists due to diversified biological activities associated with it. In view of these findings, it appeared of interest to synthesize, newer thiazolidinone derivatives with better potency.

SECTION-I

SYNTHESIS AND BIOLOGICAL EVALUATION OF 2-((Z)PHENYL)-3-(1H-PRAZOLO[3,4-b]PYRIDINE-3-YL)THIAZOLIDIN-4-ONE

With a view to getting better therapeutic agents and considering the association of various biological activities the preparation of thiazolidinone of type (IV) have been undertaken by the condensation of N-((Z)benzylidine)-1H-pyrazolo[3,4-b]Pyridine-3-amine with mercaptoacetic acid in toluene.

The constitution of the synthesized products have been characterized by using elemental analysis, infrared and $^1$H-nuclear magnetic resonance spectroscopy and further supported by mass spectroscopy.
All the compounds have been evaluated for their *in vitro* biological assay like antibacterial activity towards *Gram positive* and *Gram negative* bacterial strains and antifungal activity towards *A. niger, C. Albicans and A. Clavatus* at a different concentration. The biological activities of synthesized compounds were compared with standard drugs.
IR SPECTRAL STUDIES OF 2-(2-CHLOROPHENYL)-3-(1H-PRAZOLO[3,4-b]PYRIDINE-3-YL)THIAZOLIDIN-4-ONE

Instrument: SHIMADZU FTIR 8400 Spectrophotometer; Frequency range: 4000-400 cm\(^{-1}\) (KBr disc.)

<table>
<thead>
<tr>
<th>Type</th>
<th>Vibration Mode</th>
<th>Frequency cm(^{-1})</th>
<th>Ref.</th>
</tr>
</thead>
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<td>Observed</td>
<td>Reported</td>
</tr>
<tr>
<td>Alkane</td>
<td>C-H str. (asym.)</td>
<td>2953</td>
<td>2975-2920</td>
</tr>
<tr>
<td></td>
<td>C-H str. (sym.)</td>
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<tr>
<td></td>
<td>C-H def. (asym.)</td>
<td>1525</td>
<td>1470-1435</td>
</tr>
<tr>
<td></td>
<td>C-H def. (sym.)</td>
<td>1329</td>
<td>1395-1370</td>
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<td>C-H str.</td>
<td>3014</td>
<td>3100-3000</td>
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<td></td>
<td>C=C</td>
<td>1525</td>
<td>1585-1480</td>
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<td></td>
<td>C-H i.p. def.</td>
<td>1122</td>
<td>1125-1090</td>
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<td></td>
<td>C-H o.o.p. def.</td>
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<td>860-810</td>
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<td>C=O str.</td>
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<tr>
<td></td>
<td>S-C=N str.</td>
<td>1631</td>
<td>1640-1605</td>
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<td></td>
<td>C–N str.</td>
<td>1211</td>
<td>1220-1020</td>
</tr>
<tr>
<td></td>
<td>C–S str.</td>
<td>680</td>
<td>700-600</td>
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<td>Amine</td>
<td>N-H str.</td>
<td>3414</td>
<td>3400-3380</td>
</tr>
<tr>
<td></td>
<td>N-H def.</td>
<td>1631</td>
<td>1650-1580</td>
</tr>
</tbody>
</table>
$^1$HNMR SPECTRAL STUDIES OF 2-(2-CHLOROPHENYL)-3-(1H-PRAZOLO [3,4-b]PYRIDINE-3-YL)THIAZOLIDIN-4-ONE

Internal Standard: TMS; Solvent: CDCl$_3$ Instrument: BRUKER Spectrometer (300 MHz)

<table>
<thead>
<tr>
<th>Signal No.</th>
<th>Signal Position δppm</th>
<th>Relative No. of Protons</th>
<th>Multiplicity</th>
<th>Inference</th>
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<td>3.23-3.92</td>
<td>2H</td>
<td>multiplet</td>
<td>-CH (h)</td>
</tr>
<tr>
<td>2</td>
<td>6.41</td>
<td>1H</td>
<td>singlet</td>
<td>-CH (i)</td>
</tr>
<tr>
<td>3</td>
<td>7.12-7.15</td>
<td>1H</td>
<td>triplet</td>
<td>Ar-CH (b)</td>
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<td>7.25-7.28</td>
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<td>multiplet</td>
<td>Ar-CH (d,e,f,g)</td>
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<td>5</td>
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<td>multiplet</td>
<td>Ar-CH (c)</td>
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<tr>
<td>6</td>
<td>8.03-8.07</td>
<td>1H</td>
<td>multiplet</td>
<td>Ar-CH (a)</td>
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</tbody>
</table>
Studies on Some Heterocyclic Entities......

EXPANDED REGION

4-Thiazolidinone...
4-Thiazolidinone...
EXPERIMENTAL

SYNTHESIS AND BIOLOGICAL EVALUATION OF 2-((Z)PHENYL)-3-(1H-PRAZOLO [3,4-b]PYRIDINE-3-YL)THIAZOLIDIN-4-ONE

[A] SYNTHESIS OF N-(2-CHLOROBENZYLIDINE)-1H-PYRAZOLO[3,4-b]PYRIDINE-3-AMINE

See Part-II, Experimental Section-I [B].

[B] SYNTHESIS OF 2-(2-CHLOROPHENYL)-3-(1H-PRAZOLO[3,4-b]PYRIDINE-3-YL)THIAZOLIDIN-4-ONE

The Thiazolidinone derivatives have been synthesized by the reaction of N-(2-Chlorobenzylidene)-3H-Pyrazolo[3,4-b]pyridine-3-amine (0.01 mol, 2.56 gm) in Toluene (50.0 ml) and mercaptoacetic acid (0.02 mol, 1.84 gm) was added drop wise at room temperature in 15 min and stir for 30 min at room temperature then increase temperature slowly up to refluxed and maintain for 10 -12.0 hrs. Remove water by azeotropic distillation using a Dean-Stark separator. After completion of reaction, excess of toluene was distilled off and the resulting product was treated with 5% NaHCO₃ solution to remove unreacted mercaptoacetic acid. The separated product was washed with water, dried and recrystallized from CHCl₃ : Water. Yield 68%, m.p. 132⁰C.

Similarly other 2-((Z)phenyl)-3-(1H-prazolo[3,4-b]pyridine-3-yl)thiazolidin-4-one were prepared. Various physical constants for all the synthesized compounds are given in Table 5.

[C] BIOLOGICAL EVALUATION OF 2-((Z)PHENYL)-3-(1H-PRAZOLO [3,4-b]PYRIDINE-3-YL)THIAZOLIDIN-4-ONE

Antimicrobial testing was carried out as described in Part-II, Experimental Section-I [C]. The results of test solutions are recorded in Table 6.
### TABLE 5: PHYSICAL CONSTANTS OF 2-((Z)PHENYL)-3-(1H-PRAZOLO[3,4-b]PYRIDINE-3-YL)THIAZOLIDIN-4-ONE

<table>
<thead>
<tr>
<th>Sr. No.</th>
<th>R</th>
<th>Molecular Formula</th>
<th>Molecular Weight</th>
<th>M.P. °C</th>
<th>Yield %</th>
<th>% of Nitrogen Calcd.</th>
<th>% of Nitrogen Found</th>
<th>Rf Value</th>
<th>Solvent System</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>2-hydroxy</td>
<td>C₁₅H₁₂N₄O₂S</td>
<td>312.35</td>
<td>143</td>
<td>56</td>
<td>17.94</td>
<td>17.96</td>
<td>0.48</td>
<td>S₁</td>
</tr>
<tr>
<td>2</td>
<td>3-Nitro</td>
<td>C₁₅H₁₁N₃O₃S</td>
<td>341.34</td>
<td>148-58</td>
<td>50</td>
<td>20.52</td>
<td>20.51</td>
<td>0.54</td>
<td>S₁</td>
</tr>
<tr>
<td>3</td>
<td>4-Hydroxy</td>
<td>C₁₅H₁₂N₄O₂S</td>
<td>312.35</td>
<td>151</td>
<td>65</td>
<td>17.94</td>
<td>17.90</td>
<td>0.46</td>
<td>S₁</td>
</tr>
<tr>
<td>4</td>
<td>4-N,N-Dimethyl</td>
<td>C₁₇H₁₇N₃OS</td>
<td>339.41</td>
<td>160-63</td>
<td>60</td>
<td>20.63</td>
<td>20.65</td>
<td>0.61</td>
<td>S₁</td>
</tr>
<tr>
<td>5</td>
<td>4-Hydroxy-3-Methoxy</td>
<td>C₁₆H₁₄N₄O₃S</td>
<td>342.37</td>
<td>167-70</td>
<td>40</td>
<td>16.36</td>
<td>16.37</td>
<td>0.48</td>
<td>S₁</td>
</tr>
<tr>
<td>6</td>
<td>4-Chloro</td>
<td>C₁₅H₁ClN₂OS</td>
<td>330.79</td>
<td>134-46</td>
<td>68</td>
<td>16.94</td>
<td>16.94</td>
<td>0.46</td>
<td>S₁</td>
</tr>
<tr>
<td>7</td>
<td>4-Nitro</td>
<td>C₁₅H₁₁N₃O₃S</td>
<td>341.34</td>
<td>256</td>
<td>52</td>
<td>20.52</td>
<td>20.54</td>
<td>0.50</td>
<td>S₁</td>
</tr>
<tr>
<td>8</td>
<td>H</td>
<td>C₁₅H₁₂N₄OS</td>
<td>296.35</td>
<td>146-51</td>
<td>67</td>
<td>18.91</td>
<td>18.93</td>
<td>0.53</td>
<td>S₁</td>
</tr>
<tr>
<td>9</td>
<td>4-Methoxy</td>
<td>C₁₆H₁₄N₄O₂S</td>
<td>326.37</td>
<td>135-37</td>
<td>70</td>
<td>17.17</td>
<td>17.19</td>
<td>0.51</td>
<td>S₁</td>
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<td>10</td>
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<td>330.79</td>
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<td>16.98</td>
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<td>4-Fluoro</td>
<td>C₁₅H₁₁FN₄OS</td>
<td>314.34</td>
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<td>54</td>
<td>17.82</td>
<td>17.85</td>
<td>0.47</td>
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</tbody>
</table>

S₁  Hexane : Ethyl acetate - (7:3)
### TABLE 6: BIOLOGICAL EVALUATION OF 2-((Z)PHENYL)-3-(1H-PRAZOLO [3,4-b]PYRIDINE-3-YL)THIAZOLIDIN-4-ONE

<table>
<thead>
<tr>
<th>Sr. No.</th>
<th>Code</th>
<th>Antibacterial Activity</th>
<th>Antifungal activity</th>
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<tbody>
<tr>
<td></td>
<td></td>
<td>Minimal bactericidal concentration μg/ml</td>
<td>Minimal fungicidal concentration μg/ml</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Gram +ve Bacteria</td>
<td>Gram –ve Bacteria</td>
</tr>
<tr>
<td>1</td>
<td>3a</td>
<td>500</td>
<td>200</td>
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<tr>
<td>2</td>
<td>3b</td>
<td>500</td>
<td>250</td>
</tr>
<tr>
<td>3</td>
<td>3c</td>
<td>200</td>
<td>100</td>
</tr>
<tr>
<td>4</td>
<td>3d</td>
<td>1000</td>
<td>1000</td>
</tr>
<tr>
<td>5</td>
<td>3e</td>
<td>100</td>
<td>500</td>
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<td>6</td>
<td>3f</td>
<td>250</td>
<td>100</td>
</tr>
<tr>
<td>7</td>
<td>3g</td>
<td>500</td>
<td>200</td>
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<td>8</td>
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<td>9</td>
<td>3i</td>
<td>100</td>
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</tr>
<tr>
<td>10</td>
<td>3j</td>
<td>1000</td>
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<tr>
<td>11</td>
<td>3k</td>
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### MINIMAL INHIBITION CONCENTRATION

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<th>P.aeruginosa</th>
<th>S.aureus</th>
<th>S.pyogenus</th>
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<tr>
<td>Gentamycin</td>
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<td>0.25</td>
<td>0.5</td>
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<tr>
<td>Ampicillin</td>
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<td>100</td>
<td>250</td>
<td>100</td>
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<td>Chloramphenicol</td>
<td>50</td>
<td>50</td>
<td>50</td>
<td>50</td>
</tr>
<tr>
<td>Ciprofloxacin</td>
<td>25</td>
<td>25</td>
<td>50</td>
<td>50</td>
</tr>
<tr>
<td>Norfloxacin</td>
<td>10</td>
<td>10</td>
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</tbody>
</table>

### MINIMAL FUNGICIDAL CONCENTRATION

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<th>Standard Drugs</th>
<th>C.Albicans</th>
<th>A.Niger</th>
<th>A.Clavatus</th>
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<tbody>
<tr>
<td>Nystatin</td>
<td>100</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>Greseofulvin</td>
<td>500</td>
<td>100</td>
<td>100</td>
</tr>
</tbody>
</table>
ANTIBACTERIAL ACTIVITY:

From screening results, substituted 4-Thiazolidinone 3h (R = H) against S.aureus and 3j (R = -2-Cl) against E-coli possesses very good activity compared with ampicillin while 3e (R = -4-OH-3-OCH3) and 3i (R= -4-OCH3) against S.aureus, 3f (R= -4-Cl), 3h (R = H) and 3i (R= -4-OCH3) against S.pyogenus possesses moderate activity while 3g (R= -4-NO2) and 3j (R = -2-Cl) possesses moderate activity against E-coli and P.aeruginosa respectively as compared with ampicillin. The remaining 4-Thiazolidinone derivatives possess poor activity against all four bacterial species.

ANTIFUNGAL ACTIVITY:

Antifungal screening data showed that substituted 4-Thazolidinone 3f (R = -4-Cl) and 3j (R = -2-Cl) show highly promising activity against C. albicans as compare to Greseofulvin while 3d (R = -4-N,N(CH3)2) & 3i (R = -4-OCH3) against A.niger and also 3f (R = -4-Cl) against A.clavatus show good activity compare with standard drug. The remaining compounds exhibited only moderate to poor activity.
REFERENCES


4-Thiazolidinone...


