SYNTHESIS OF 5-{(4-AMINO-N-[2-(DIETHYLAMINO)ETHYL]-
-o-ANISAMIDO-5-YL}-AMINO-3-SUBSTITUTEDIMINO-7-
-SUBSTITUTEDIMINO-1,2,4,6-TRITHIAZEPINES.

D. T. Tayade¹, R. D. Thombare¹, S. A. Waghmare²

¹Department of Chemistry, Government Vidarbha Institute of Science and Humanities, Amravati 444606.
²Department of Chemistry, Ghulam Nabi Azad Arts, Comm. & Science College, Barshitakli, Dist. Akola 444401.

Abstract:
A novel series of 5-{(4-amino-N-[2-(diethylamino)ethyl]-o-anisamido-5-yl]-amino-3-substitutedimino-7-
substitutedimino-1,2,4,6-trithiazepines was synthesized by the interactions of 4-amino-5-substituteddithiobiureto-N-
[2-(diethylamino)ethyl]-o-anisamides with phenylisothiocarbamoyldichloride in acetone-ethanol medium. The
structures of all the synthesized compounds were justified on the basis of chemical characteristics, elemental
analysis and spectral studies.

Keywords: 5-{(4-Amino-N-[2-(diethylamino)ethyl]-o-anisamido-5-yl]-amino-3-phenylimino-7-ethylimino-1,2,4,6-
trithiazepine, 4-amino-5-phenylthiobiureto-N-[2-(diethylamino)]-ethyl]-o-anisamide, phenylisothiocarbamoyldichloride, acetone-ethanol medium.

Corresponding author:
D.T. Tayade,
Department of Chemistry,
Government Vidarbha Institute of Science and Humanities,
Amravati 444606.
Email: skdtayade@gmail.com,
rupalidthombare30@gmail.com.

Please cite this article in press as D.T. Tayade et al., Synthesis of 5-{(4-Amino-N-[2-(Diethylamino) Ethyl] – o -
Anisamido-5-YL] – Amino – 3 – Substitutedimino – 7 –Substitutedimino - 1, 2, 4, 6- Trithiazepines, Indo Am. J. P.
INTRODUCTION:
The literature survey reveals that heterocyclic compounds are used as drugs. It has been reported that the thiocarbamides exhibit antibacterial [1], fungicidal [2] insecticidal [3], antiviral [4], anesthetic [5] and have many biological activities. The most remarkable application of thiocarbamide is used as commercial pesticides, particularly herbicides [6-10]. Acyclic thiocarbamides were used as an intermediate for the synthesis of thia triazepines. Recently we have synthesized 4-amino-5-substituted dithiobiureto-N-[2-(diethylamino)ethyl]-o-anisamides. Due to significance of thia triazepines in agricultural, medicinal, industrial and pharmaceutical sciences, it was thought interesting to carry out cyclisation of 4-amino-5-substituted dithiobiureto-N-[2-(diethylamino)ethyl]-o-anisamides in a new type of thia triazepines.

In the present work 5-{4-amino-N-[2-(diethylamino)ethyl]-o-anisamido-5-yl}-amino-3-substitutedimino-7-substitutedimino-1,2,4,6-trithiazepines was synthesized by the interactions of 4-amino-5-substituted dithiobiureto-N-[2-(diethylamino)ethyl]-o-anisamides with phenylisothiocarbarnoyleyl dichloride in acetone-ethanol medium. The probable reaction and mechanism is depicted below (Scheme-VI).

![Scheme-VI](image-url)
Scheme - VI
R= t-butyl, phenyl, p-chlorophenyl, Ethyl, methyl, o-tolyl, m-tolyl, p-tolyl

Synthesis of 5-{(4-amino -N-[2-(diethylamino)ethyl] -o-anisamido-5-yl) -amino-3-phenylimino -7- ethylimino-1, 2, 4, 6-trithiazepine}
5- {(4- Amino N-[2- (diethylamino)ethyl] -o-anisamido-5-yl)- amino -3- phenylimino -7- ethylimino-1,2,4,6-trithiazepine was synthesized by the interaction of 4-amino -5- ethyldithiobiureto -N-[2-(diethylamino) ethyl] -o- anisamide and phenylisothiocarbamoyl- chloride in acetone-ethanol medium by refluxing on water bath for 2 hours. The reaction mixture was filtered in hot conditions. After distillation of excess solvent brownish yellow crystals were isolated, on basification with ammonia it gave 5- {(4-amino-N-[2- (diethylamino) ethyl] -o-anisamido-5-yl) -amino -3- phenylimino-7- ethylimino-1,2,4,6-trithiazepine. Yield 90%, m.p.223°C.

The probable reaction and mechanism depicted below,

Reaction

4-Amino-5-ethyldithiobiurato-N-[[2-diethylamino)-ethyl]-o-anisamide

Phenyldichloroisothiocyanate

Acetone
Reflux

5-{4-Amino-N-[2-(diethylamino)-ethyl]-o-anisamido-5-yl}-amino-3-phenylimino-7-ethylimino-1,2,4,6-trithiazepine
Properties:
It is faint yellow crystalline solid having M. P. 245°C. It gave positive test for nitrogen and sulphur. It does not desulphurized when boiled with sodium plumbite solution which clearly indicates that sulphur is not free and gets cyclised [11, 12]. Soluble in benzene, DMF, acetic acid and acetone. It forms picrate having m.p. 250°C.

Elemental Analysis:
This result of elemental analysis is gives Carbon [52.77% (found), 53.66% (calculated)], Hydrogen [05.00 % (found), 05.90% (calculated)], Nitrogen [16.80 % (found), 17.53% (calculated)], Sulphur [16.17% (found), 17.17% (calculated)]. From the analytical data the molecular formula was found to be C_{23}H_{33}N_{7}O_{2}S_{3}.

IR Spectrum:
The IR spectrum of compound was carried out in KBr pellets, the important absorption are correlated as (cm⁻¹) 3390.10 N-H stretching, 2927.20 C-H stretching, 1644.21 C=O stretching, 1338.21 C-N stretching, 1154.13 C=S stretching, 0666.26 C-S stretching.

PMR Spectrum:
The PMR spectrum of the compound was carried out in CDCl₃ and DMSO-d₆. This spectrum distinctly displayed the signals due to Ar-H protons at δ 8.600 ppm, Ar-H (phenyl) protons at δ 6.4836 ppm, -NH proton at δ 5.4228-5.1084 ppm, NH₂ protons at δ 4.9117-4.0160 ppm, -OCH₃ protons at δ 3.2993 ppm, CH₂ protons at δ 2.5174-2.0896 ppm, N-CH₃ protons at δ 1.2368 ppm and -CH₃ protons at δ 0.9778 ppm. From the above properties and spectral analysis of the compound was assigned the structure as 5-[(4-amino-N-[2-(diethy lamino) ethyl]-o-anisamido-5-yl)-amino-3-phenyl-imino-7-ethylimino-1, 2, 4, 6-trithiazepines.
Table No. I-1

<table>
<thead>
<tr>
<th>Sr. No.</th>
<th>Expt. No.</th>
<th>5-{(4-Amino-N-[2-(diethylamino)ethyl]-o-anisamido-5-yl]-amino-3-substituted-imino-7-substitutedimino-1,2,4,6-trithiazepines</th>
<th>Yield (%)</th>
<th>m.p. (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>(IIIb)</td>
<td>5-{(4-Amino-N-[2-(diethylamino)ethyl]-o-anisamido-5-yl]-amino-3-phenylimino-7-methylimino-1,2,4,6-trithiazepine</td>
<td>80</td>
<td>219</td>
</tr>
<tr>
<td>2</td>
<td>(IIIc)</td>
<td>5-{(4-Amino-N-[2-(diethylamino)ethyl]-o-anisamido-5-yl]-amino-3-phenylimino-7-t-butylimino-1,2,4,6-trithiazepine</td>
<td>85</td>
<td>221</td>
</tr>
<tr>
<td>3</td>
<td>(IIId)</td>
<td>5-{(4-Amino-N-[2-(diethylamino)ethyl]-o-anisamido-5-yl]-amino-3-phenylimino-7-p-Chlorophenylimino-1,2,4,6-trithiazepine</td>
<td>90</td>
<td>227</td>
</tr>
<tr>
<td>4</td>
<td>(IIIe)</td>
<td>5-{(4-Amino-N-[2-(diethylamino)ethyl]-o-anisamido-5-yl]-amino-3-phenylimino-7-o-tolylimino-1,2,4,6-trithiazepine</td>
<td>92</td>
<td>230</td>
</tr>
<tr>
<td>5</td>
<td>(IIIf)</td>
<td>5-{(4-Amino-N-[2-(diethylamino)ethyl]-o-anisamido-5-yl]-amino-3-phenylimino-7-m-tolylimino-1,2,4,6-trithiazepine</td>
<td>94</td>
<td>233</td>
</tr>
<tr>
<td>6</td>
<td>(IIIg)</td>
<td>5-{(4-Amino-N-[2-(diethylamino)ethyl]-o-anisamido-5-yl]-amino-3-phenylimino-7-p-tolylimino-1,2,4,6-trithiazepine</td>
<td>89</td>
<td>240</td>
</tr>
</tbody>
</table>

REFERENCES: