Synthesis Of 5-{(4-Amino-N-[2-(Diethylamino)Ethyl]-O-Anisamido-5-Yl}-Amino-3-Substitutedimino-7-Substitutedimino-1,2,4,6-Trithiazepines

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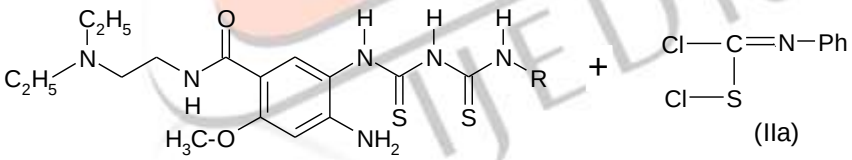
Abstract - A novel series of 5-{(4-amino-N-[2-(diethylamino)ethyl]-o-anisamido-5-yl}-amino-3-substitutedimino-7-substitutedimino-1,2,4,6-trithiazepines was synthesized by the interactions of 4-amino-5-substituteddithiobiureto-N-[2-(diethylamino)ethyl]-o-anisamides with phenylisothio carbamoyldichloride 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-phenyldithiobiureto-N-[2-(diethylamino) ethyl]-o-anisamide, phenylisothiocarbamoylchloride, acetone-ethanol medium.

I. 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 thiocabamides were used as an intermediate for the synthesis of thiatriazepines. Recently we have synthesized 4-amino-5-substituteddithiobiureto-N-[2-(diethylamino) ethyl]-o-anisamides. Due to significances of thiatriazepines in agricultural, medicinal, industrial and pharmaceutical sciences, it was thought interesting to carry out cyclisation of 4-amino-5-substituteddithiobiureto-N-[2-(diethylamino)ethyl]-o-anisamides in a new type of thiatriazepines**.**

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 phenylisothiocabamoyldichloride in acetone-ethanol medium. The probable reaction and mechanism is depicted below (**Scheme-VI)**,



(Ia)

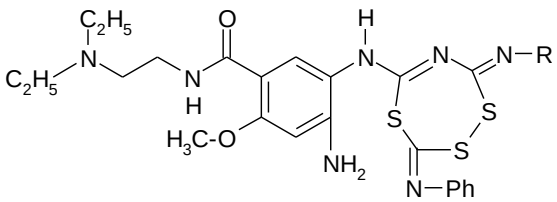
4-Amino-5-substituteddithiabiurato-N-[(2-diethylamino)-ethyl]-o-anisamide

(IIa)

Phenyldichloroisothiocyanate

Acetone

Reflux



(IIIa)

5-{4-Amino-N-[2-(diethylamino)-ethyl]-o-anisamido-5-yl}-amino-3-phenylimino-7-substitutedimino-1,2,4,6-trithiazepines

Scheme- VI

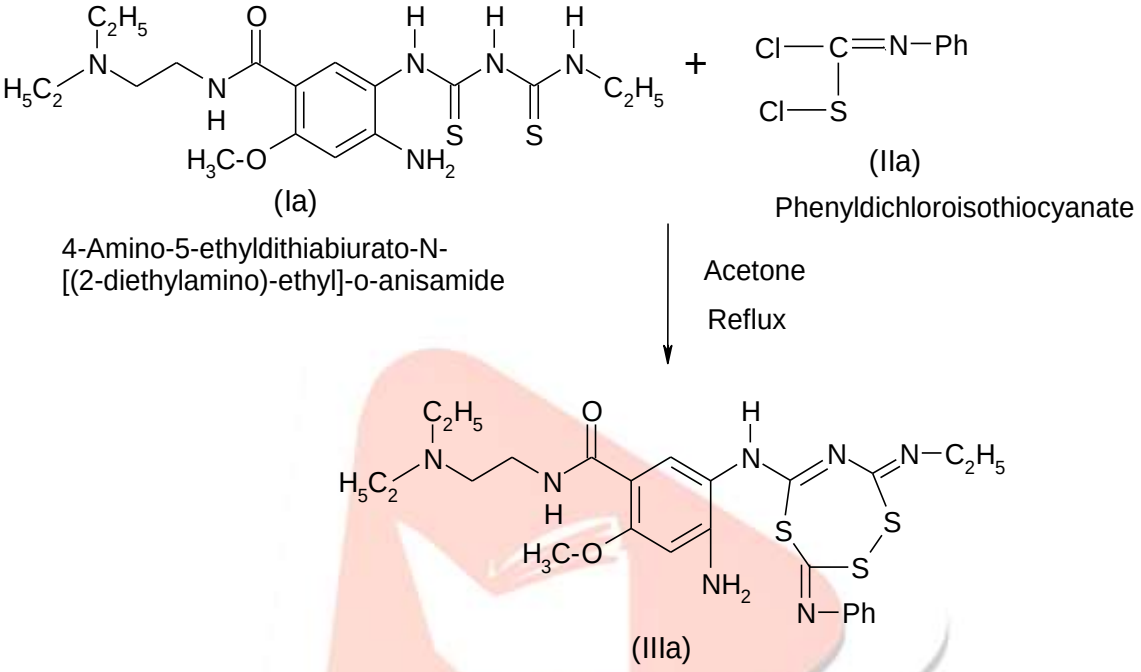
R= t-butyl, phenyl, p-chlorophenyl, Ethyl, methyl, o-tolyl, m-tolyl, p-tolyl

II. 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 sovent 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

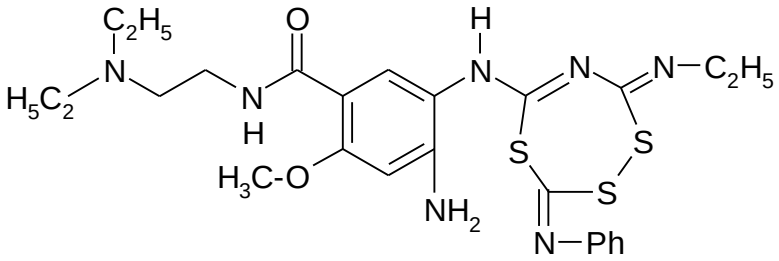


Properties

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 $C_{25}H_{33}N_7O_2S_3$. **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 $CDCl_3$ and DMSO-d_6**.** This spectrum distinctly displayed the signals due to Ar-H protons at δ 8.6000 ppm, Ar-H (phenyl) protons at δ 6.4836 ppm,–NH proton at δ 5.4228-5.1084 ppm, NH_2 protons at δ 4.9117-4.0160 ppm, -OCH_3 protons at δ 3.3993 ppm, CH_2 protons at δ 2.5174-2.0896 ppm, N-CH_3 protons at δ 1.2368 ppm and -CH_3 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-(diethylamino)ethyl]-o-anisamido-5-yl]-amino-3-phenyl-imino-7-ethylimino-1,2,4,6-trithiazepines **.**



(IIIa)

5-{4-Amino-N-[2-(diethylamino)-ethyl]-o-anisamido-5-yl}-amino-3-phenylimino-7-ethylimino-1,2,4,6-trithiazepine

Similarly, 4-amino-5-methyldithiobiureto-N-[2-(diethylamino)ethyl]-o-anisamide **(Ib),** 4-amino-5-t-butyldithiobiureto-N-[2-(diethylamino)ethyl]-o-anisamide **(Ic),** 4-amino-5-p-chlorophenyldithiobiureto-N-[2-(diethylamino)ethyl]-o-anisamide **(Id),** 4-amino-5-o-tolyldithiobiureto-N-[2-(diethylamino)ethyl]-o-anisamide **(Ie),** 4-amino-5-m-tolyldithio- biureto-N-[2-(diethylamino)ethyl]-o-anisamide **(If),** 4-amino-5-p-tolyldithiobiureto-N-[2-(diethylamino)ethyl]-o-anisamide **(Ig)** with phenylisothiocarbamoyldichloride **(IIa)** in acetone-ethanol medium were refluxed on water bath to isolate the respective 5-{(4-amino-N-[2-(diethylamino)ethyl]-o-anisamido-5-yl]-amino-3-phenylimino-7-methyl-imino-1,2,4,6-trithiazepines **(IIIb)** 5-{(4-amino-N-[2-(diethylamino)ethyl]-o-anisamido-5-yl}-amino-3-phenylimino-7-t-butylimino-1,2,4,6-trithiazepines **(IIIc)** 5-{(4-amino-N-[2-(diethylamino)ethyl]-o-anisamido-5-yl}-amino-3-phenylimino-7-p-chlorophenylimino -1,2,4,6-trithi- azepines **(IIId)** 5-{(4-amino-N-[2-(diethylamino)ethyl]-o-anisamido-5-yl}-amino-3-phenylimino-7-o-tolylimino-1,2,4,6-trithiazepines **(IIIe)** 5-{(4-amino-N-[2-(diethylamino)ethyl]-o-anisamido-5-yl}-amino-3-phenylimino-7-m-tolylimino-1,2,4,6-trithiazepines **(IIIf)** 5-{(4-amino-N-[2-(diethylamino)ethyl]-o-anisamido-5-yl}-amino-3-phenylimino-7-p-tolylimino-1,2,4,6-trithiazepines **(IIIg)** by above mentioned methods as described in Experiment No. 3 -8 listed in **Table No. I-1**

Table No. I-1

Sr. No.	**Expt. No.**	5-{(4-Amino-N-[2-(diethylamino)ethyl]-o-anisamido-5-yl}-amino-3-**substituted-imino**-7-**substitutedimino**-1,2,4,6-trithiazepines	**Yield (%)**	**m.p. (^{0}C)**
1	**(IIIb)**	5-{(4-Amino-N-[2-(diethylamino)ethyl]-o-anisamido-5-yl}-amino-3-**phenylimino** - 7-**methylimino**-1,2,4,6-trithiazepine	**80**	**219**
2	**(IIIc)**	5-{(4-Amino-N-[2-(diethylamino)ethyl]-o-anisamido-5-yl}-amino-3-**phenylimino** - 7-**t-butylimino**-1,2,4,6-trithiazepine	**85**	**221**
3	**(IIId)**	5-{(4-Amino-N-[2-(diethylamino)ethyl]-o-anisamido-5-yl}-amino-3-**phenylimino** - 7-**p-Chlorophenylimino**-1,2,4,6-trithiazepine	**90**	**227**
4	**(IIIe)**	5-{(4-Amino-N-[2-(diethylamino)ethyl]-o-anisamido-5-yl}-amino-3-**phenylimino** - 7-**o-tolylimino**-1,2,4,6-trithiazepine	**92**	**230**
5	**(IIIf)**	5-{(4-Amino-N-[2-(diethylamino)ethyl]-o-anisamido-5-yl}-amino-3-**phenylimino** - 7-**m-tolylimino**-1,2,4,6-trithiazepine	**94**	**233**
6	**(IIIg)**	5-{(4-Amino-N-[2-(diethylamino)ethyl]-o-anisamido-5-yl}-amino-3-**phenylimino** - 7-**p-tolylimino**-1,2,4,6-trithiazepine	**89**	**240**

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