



CODEN [USA]: IAJPBB

ISSN: 2349-7750

INDO AMERICAN JOURNAL OF
PHARMACEUTICAL SCIENCES

<http://doi.org/10.5281/zenodo.1067612>

Available online at: <http://www.iajps.com>

Research Article

**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. Tayade^{1*}, 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 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.

Corresponding author:

D.T.Tayade,

Department of Chemistry,
Government Vidarbha Institute of Science and Humanities,
Amravati 444606.

Email:- skdtayade@gmail.com,
rupalidhombare30@gmail.com.



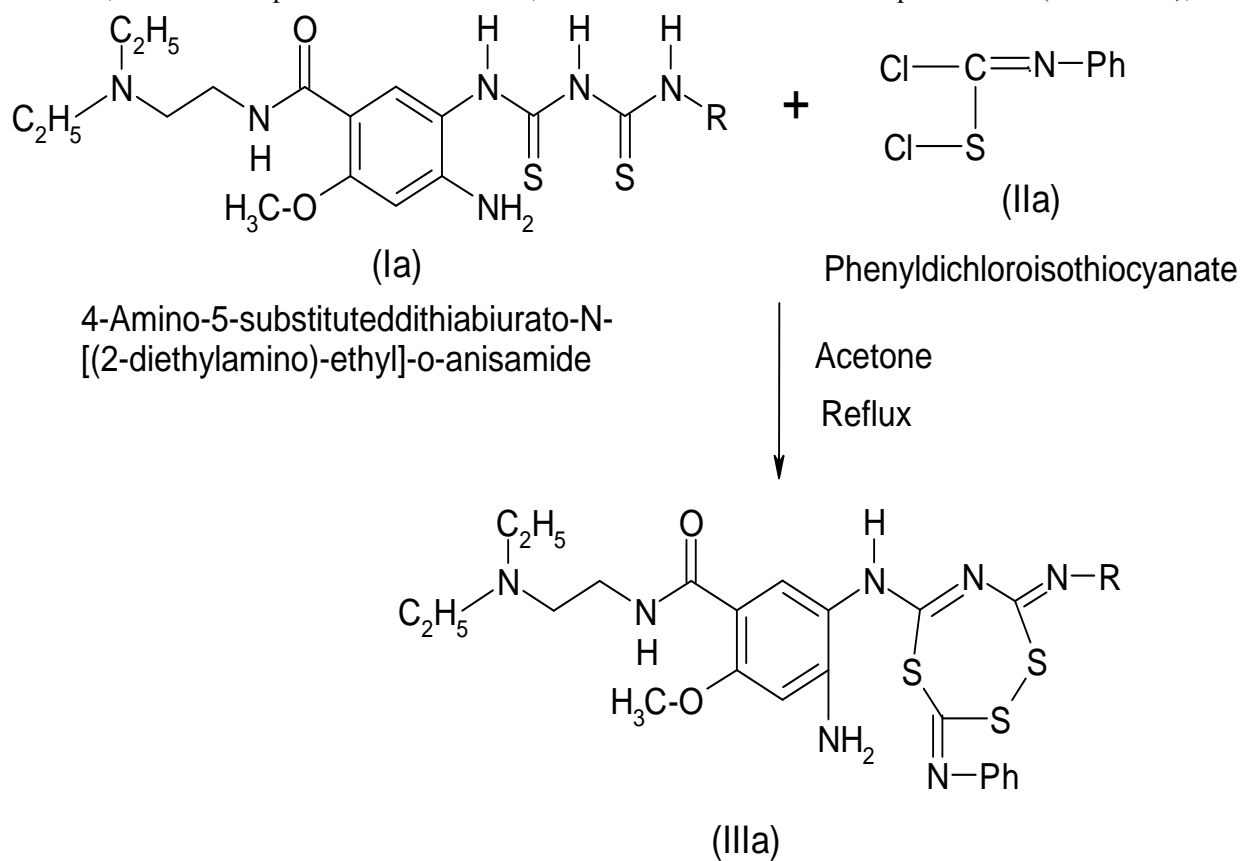
Please cite this article in press as D.T.Tayade et al., *Synthesis of 5- {{(4-Amino-N- [2- (Diethylamino) Ethyl] – o - Anisamido-5-YL} – Amino – 3 – Substitutedimino – 7 – Substitutedimino - 1, 2, 4, 6- Trithiazepines, Indo Am. J. P. Sci, 2017; 4[11].*

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 thiazepines. Recently we have synthesized 4-amino-5-substituted dithiobiureto-N-[2-(diethylamino)ethyl]-o-anisamides. Due to significances of thiazepines 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 thiazepines.

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



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

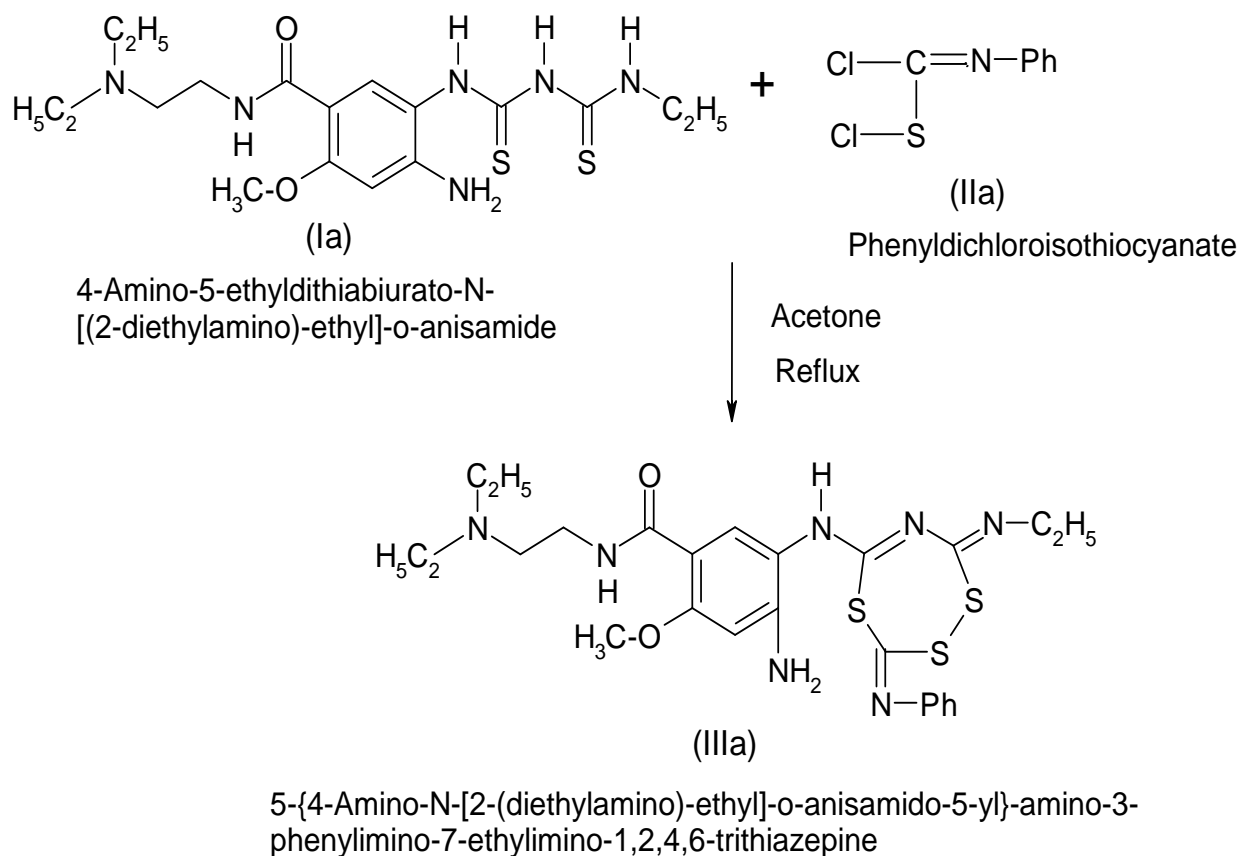
Scheme- VI

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-ethylthiobiureto-N-[2-(diethylamino)ethyl]-o-anisamide and phenylisothiocarbonyl-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

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 C₂₅H₃₃N₇O₂S₃.

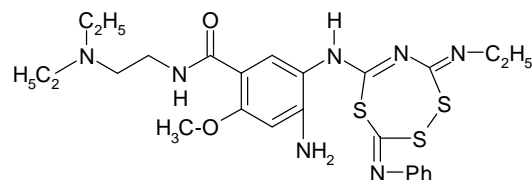
IR Spectrum:

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 8.6000 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 3.3993 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-(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- methylthiobiureto -N- [2-(diethylamino) ethyl] -o- anisamide (**IIb**), 4- amino -5-t- butyldithiobiureto -N- [2-(diethylamino)ethyl] -o- anisamide (**IIc**), 4- amino -5 -p-chlorophenyldithiobiureto -N- [2-(diethylamino) ethyl] -o- anisamide (**IId**), 4- amino -5-o- tolyldithiobiureto -N- [2- (diethylamino) ethyl] -o- anisamide (**IIe**), 4- amino -5-m- tolyldithio- biureto -N- [2- (diethylamino) ethyl] -o- anisamide (**IIIf**), 4- amino-5-p-tolyldithiobiureto -N- [2-(diethylamino) ethyl] -o- anisamide (**IIg**) with phenylisothiocarbonyldichloride (**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 (**IIIIf**) 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**

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. (°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	(III d)	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	(III f)	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	(III g)	5-[(4-Amino-N-[2-(diethylamino)ethyl]-o-anisamido-5-yl)-amino-3-phenylimino -7-p-tolylimino-1,2,4,6-trithiazepine	89	240

REFERENCES:

1. Tayade D.T., Proc., 83rdInd. Sci. Cong., 1996.
2. Mota J. F., Manuel J., Fernandez G., Mellet C.O. and Adril M.A.P., Carbohy.res, 188,1989, 35-44.
3. Oudir B., Rigo, J.P., Hénichart P., Gautret, Synthesis, 2006, 2845-2848.
4. Beazley B., Moss S.F., Pritchard R.G., Taylor D.R., Acta Cryst, 37, 1981, 486.
5. Taylor D.R., Moss S.F., J.Chem. Soc, Perkin Trans, 1, 1982, 1999 – 2005.
6. Cablewski T., Forsyth C.M., Francis C.L., Liepa A.J., Tran. Aust. J. chem, 61(10), 2010, 785-796.
7. Murai N., Komatsu M., Ohshiro Y., azawa T., chemistry Letter, 1976, 1379-80.
8. Dunn P.J. and Ress C.W., J. Chem.Soc, Perkin Trans, 1, 1989, 1405-1410.
9. Dunn P.J., Morris J.L. and Ress C.W., J. Chem.Soc, Perkin Trans,1988, 1745-48.
10. Taylor D.R. and Moss S.F., J. Chem.Soc, Chem.Comm, 1980,156.
11. Hector D. S., Ber,25, 1992.779.
12. Hector D.S., Oefvers Kong Vet.Akad., 89, 1992.