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Research Article

**SYNTHESIS OF 2-SUBSTITUTEDIMINO-6-*o*-TOLYLIMINO-4-AMINO
[(2-*o*-TOLYLTHIOCARBAMIDO)1,3-BENZOTHIAZOLO]1,3,5-
DITHIAZINE****K. S. Panpaliya^{1*}, D. T. Tayade¹, R. D. Isankar¹, S. S. Padhen²**¹Department of Chemistry, Government Vidarbha Institute of Science and Humanities, Amravati 444604.²Department of Chemistry, R.S. Science College, Chandur (Railway) 444 904.**Abstract:**

Recently a series of 2-substitutedimino-6-*o*-tolylimino-4-amino[(2-*o*-tolyl thiocarbamido)-1,3-benzothiazolo]-1,3,5-dithiazines had been synthesized by refluxing 2-(*N'*-*o*-tolyl)-thiocarbamido-5-(*N''*-*o*-tolyl)-dithiabiurato-1,3-benzothiazoles with various isocyanodichlorides in 60% acetone-ethanol medium. The structures of all synthesized compounds were justified on the basis of chemical characteristics, elemental analysis and spectral studies.

Keywords: Various isocyanodichlorides, 2-(*N'*-*o*-tolyl)thiocarbamido-5(*N''*-*o*-tolyl)2,4-dithiabiurato-1,3-benzothiazoles.

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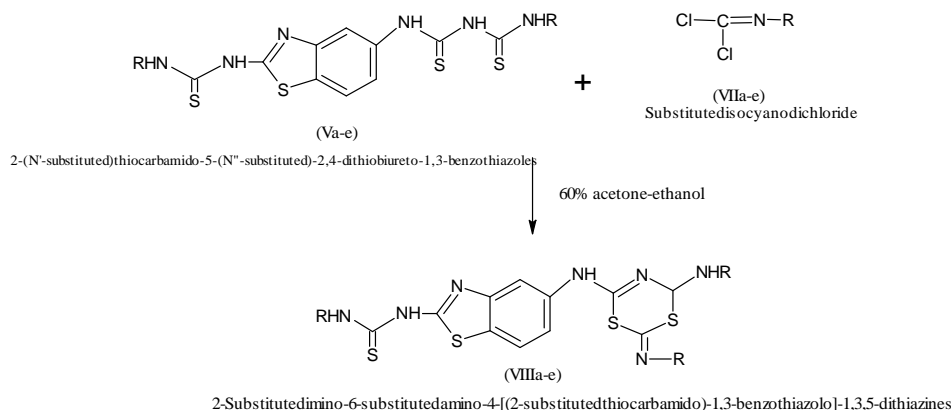
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INTRODUCTION:

In the field of organic chemistry heteroacyclic and heterocyclic compounds have enormous important in various fields. It is reported that heterocyclic compounds containing 1,3,5-dithiazino and 1,3,5-thiadiazino nucleus are widely used in multidisciplinary fields [1-5]. 1,3,5-Dithiazino and 1,3,5-thiadiazino nucleus containing molecules have drug like properties [6-14] and are used pharmaceutical and industrial fields. Literature survey reveals that dithiazine nucleus possesses anti-helminthic [15], fungicidal [16], insecticidal [17], antibacterial [18], anti-tuberculostatic [19], anti-cancer [20] antiviral [21] and anti-HIV [22] properties. Dithiazine and its derivatives possess agriculture [23] as well as industrial [24] applications and are effective against copper corrosion²⁴,

lubricating oil [25]. Few researchers [26] had been briefly urbanized significant reactions of substituted isocyanodichlorides and its derivatives.

As a wider program of this laboratory in synthesis of nitrogen, nitrogen and sulphur containing heteroacycles and heterocycles to develop an alternative route for synthesis of 5,6 and 7 member heterocycles, it is quite interesting to interact 2-(N'-*o*-tolyl)thiocarbamido-5-(N''-*o*-tolyl)-2,4-dithiobiurato-1,3-benzothiazole(**Va-e**) with N-substitutedisocyanodichlorides(**VIIa-e**) in 60% acetone-ethanol medium to isolate yet new series 2-substitutedimino-6-*o*-tolylimino-4-[(2-*o*-tolylthiocarbamido)-1,3-benzothiazolo]-1,3,5-dithiazine (**VIIIa-e**).



Where, R=ethyl, -methyl, -phenyl, -tert-butyl, -p-chlorophenyl, -*o*-tolyl.

MATERIALS AND METHOD:

All chemicals used were of Mercks Millipore (Indian made).

Method

Method employed in the present experiments for the synthesis of various substituted dithiobiurate is conventional refluxing under water bath for different hours for different experiments.

Experimental

The melting points of synthesized compounds were recorded using hot paraffin bath. The carbon and hydrogen analysis was carried out on Carlo-Ebra-1106 analyzer. Nitrogen estimation was carried out on Colman-N-analyzer-29. IR spectra were recorded on Perkin Elmer spectrometer in the range 4000-400 cm⁻¹ in KBr pellets. PMR spectra were recorded on BRUKER AVANCE II 400 NMR spectrometer with TMS as an internal standard using CDCl₃ and DMSO-d₆ as a solvent. The purity of the compounds

were checked on silica gel -G plates by TLC with layer thickness of 3mm.

RESULTS AND DISCUSSION:**1) General procedure for synthesis of 2-*o*-tolylimino-6-*o*-tolylimino-4-amino [(2-*o*-tolylthiocarbamido) 1, 3-benzothiazolo]-1, 3, 5-dithiazine (VIIIa1)**

An interaction of (N'-*o*-tolyl) thiocarbamido-5-(N''-*o*-tolyl)-2,4-dithiobiurato-1,3-benzothiazole (**Va**) and *o*-tolylisocyanodichloride(**VIIa**) was carried out in 60% acetone-ethanol medium for 4 hours on water bath. During refluxing evolution of hydrochloride gas was clearly noticed. After distillation of excess solvent, ivory colour product was isolated which on basification with dilute ammonium hydroxide gave yellow crystals of 2-*o*-tolylimino-6-*o*-tolylimino-4-[(2-*o*-tolylthiocarbamido)1,3-benzothiazolo]-1,3,5-dithiazine (**VIIIa**).

Spectral Characterization of synthesized compound

Synthesis of 2-*o*-tolylimino-6-*o*-tolylimino-4-amino [(2-*o*-tolylthiocarbamido) 1, 3-benzothiazolo]-1, 3, 5-dithiazine (VIIIa)

It is a Yellow brownish crystalline solid, molecular formula $C_{32}H_{27}N_7S_4$, Yield-88%, M.P. 184°C, %Composition found(calculated): C-55.60 (56.61), H-3.20 (4.23), N-15.38(15.38) and S-19.21 (20.09); FTIR (KBr) ν cm^{-1} - 3329 (N-H stretching), 3064(Ar-CH stretching), 2189 (S-C=N stretchinh), 1645(C=NH stretching), 1531 (C=C stretching), 1402 (-C=N stretching), 1134 (C-N stretching) and 743 (C-S stretching); 1H NMR (400 MHz $CDCl_3$ δ ppm) This spectrum distinctly displayed the signals due to -NH proton flanked inthioamido and benzothiazole ring at δ 8.6682 ppm,-NH proton flanked in thiamido and phenyl ring at δ 8.2977-8.2222 ppm, Ar-H proton of benzothiazole ring at δ 7.7365-7.0688 ppm, Ar-H protons singlet at 6.9821-6.7538 ppm,-CH₃ proton singlet at δ 1.2371 ppm. ^{13}C NMR (400 MHz $CDCl_3$ δ ppm) This spectrum distinctly displayed the

signals due to C=S carbon at δ 181.72-169.11 ppm, Ar-C carbon at δ 139.66-111.70 ppm, -CH₃ carbon at δ 19.43 ppmrespectively; LC-MS (m/z) Mol. Wt.: 640.

Similarly,2-(N'-*o*-tolyl)-thiocarbamido-5-(N''-*o*-tolyl)-2,4-dithiobiurato-1,3-benzothiazole(Va) interacted with ethyl isocyanodichloride (VIIb),tert-butylisocyanodichloride (VIIc), *p*-chlorophenylisocyanodichloride (VIIId) and phenylisocyanodichloride (VIIe) by above mentioned method to isolate 2-ethylimino-6-*o*-tolylimino-4-amino-[(2-*o*-tolylthiocarbamido)-1,3-benzothiazolo]-1,3,5-dithiazine (VIIIb),2-tert-butylimino-6-*o*-tolylimino-4-amino-[(2-*o*-tolylthiocarbamido)-1,3-benzothiazolo]-1,3,5-dithiazine (VIIIc), 2-*p*-chlorophenylimino-6-*o*-tolylimino-4-amino-[(2-*o*-tolylthiocarbamido)-1,3-benzothiazolo]-1,3,5-dithiazine(VIIId), 2-phenylimino-6-*o*-tolylimino-4-amino-[(2-*o*-tolylthiocarbamido)-1,3-benzothiazolo]-1,3,5-dithiazine (VIIIE) respectively and enlisted in Table No.1.

Table No.1

Sr No	Compd. No	2-Substitutedimino-6- <i>o</i> -tolylimino-4-amino[(2- <i>o</i> -tolylthiocarbamido)1,3-benzothiazolo]-1,3,5-dithiazine(VIIIb-e)	Yield (%)	m.p °C
1	[VIIIb]	2-Ethyl-----6- <i>o</i> -toylimino-----1,3,5-dithiazine	81	229
2	[VIIIc]	2-tert-Butyl-----6- <i>o</i> -toylimino-----1,3,5-dithiazine	77	250
3	[VIIId]	2- <i>p</i> -Chlorophenyl---6- <i>o</i> -toylimino---1,3,5-dithiazine	69	267
4	[VIIIE]	2-Phenyl-----6- <i>o</i> -toylimino-----1,3,5-dithiazine	85	290

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