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Research Article

**SYNTHESIS OF
1-O-TOLYL-3-SUBSTITUTED-2,6-DITHIO-4-AMINO
-[(2-O-TOLYLTHIOCARBAMIDO)-1,3-BENZOTHIAZOLO]
-1,3,5-TRIAZINE****K. S. Panpaliya^{1*}, D. T. Tayade², R. S. Shaikh³**¹Department of Chemistry, Government Vidarbha Institute of Science and Humanities, Amravati 444 604, Maharashtra State, India. E-mail- krish16591@gmail.com²Department of Chemistry, Government Vidarbha Institute of Science and Humanities, Amravati 444 604, Maharashtra State, India. E-mail: skdtayade@gmail.com³Department of Chemistry, Government Vidarbha Institute of Science and Humanities, Amravati 444 604, Maharashtra State, India. E-mail: rahimgvish@gmail.com**Abstract:**

We reported synthesis of 1-o-tolyl-3-substituted-2,6-dithio-4-amino-[(2-o-tolylthiocarbamido)-1,3-benzothiazolo]-1,3,5-triazines by isomerisation of 2-substituted-imino-6-o-tolylimino-4-[(2-o-tolylthiocarbamido)-1,3-benzothiazolo]-1,3,5-dithiazines in 10 % aqueous ethanolic sodium bicarbonate mixture in ethanol solvent. The structure determination and justification of the synthesized compounds were done on the basis of elemental analysis, chemical characteristics and spectral studies.

Keywords: 2-substituted-imino-6-o-tolylimino-4-[(2-o-tolylthiocarbamido)-1,3-benzothiazolo]-1,3,5-dithiazines, Isomerization, Sodium bicarbonate, Ethanol.

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INTRODUCTION:

Heterocyclic compounds containing nitrogen and sulphur in ring became selective drug due to their versatile significances [1,2] and applications in biotechnological, pharmaceutical, medicinal, biochemical, agricultural and industrial sciences. The 1,3,5-triazine in heterocyclic system showed number of bioactive molecules such as herbicides and pharmaceuticals products [3]. s-Triazino molecules are known for their anti-cancer [4], anti-HIV [5], anti-malarial [6], antimicrobial [7], antiviral [8], anti-trypanosomal [9], hypolipidemic activities [10], estrogen receptors modulators [11], sorbitol dehydrogenase inhibitors [12] and VLA-4 integrin antagonists [13]. Kodape et al [14] synthesized hematological and behavioral studies of fresh water fish, *Channa Orientalis*(Sch) exposed to novel derivative of 1,3,5-triazine, Waghmare and Tayade¹⁵ synthesized 5-substituted derivatives of 2,4-dithio-3-phenyl-6-chalcone-1,3,5-triazines. Hence, it was thought interesting to synthesize 1-o-tolyl-3-substituted-2,6-dithio-4-amino-[(2-o-tolylthiocarbamido)-1,3-benzothiazolo]-1,3,5-triazines (IIa-e) by an isomerisation of 2-substituted imino-6-o-tolylimino-4-amino-[(2-o-tolylthiocarbamido)-1,3-benzothiazolo]-1,3,5-dithiazines (Ia-e) respectively in 10% ethanolic sodium bicarbonate solution.

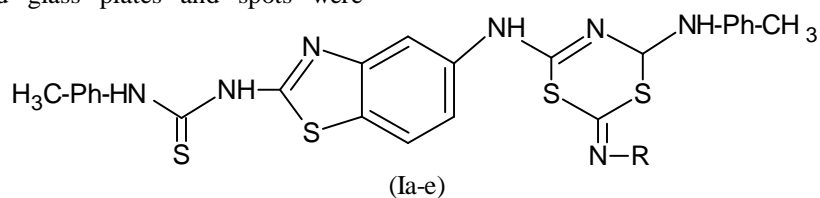
MATERIALS AND METHOD:

AR grade Merck and Sigma chemicals were used for synthesis. Follow-up of reactions and checking homogeneity of compounds were made by TLC on silica Gel-protected glass plates and spots were

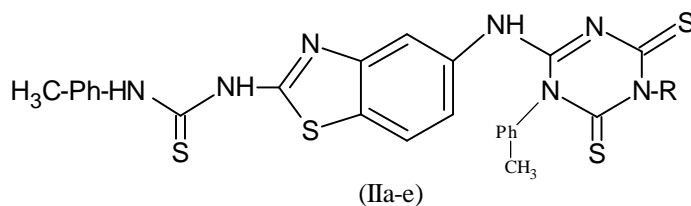
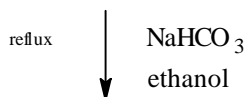
detected by exposure to UV-lamp at 1254 and 1365 nm. Unless otherwise noted. Carbon and hydrogen analysis was carried out on Carlo-Ebra-1106 analyser. Nitrogen estimations were carried out on Colman-N-analyser-29, while sulphur estimations were carried out by Carius method. Melting points of all compounds were determined in open glass capillaries with a SGW X-4 digital apparatus and were uncorrected. IR spectra were recorded on Perkin-Elmer spectrometer in range of 4000-400 cm⁻¹ in KBr pellets. ¹H NMR and ¹³C NMR spectra were recorded on Bruker ARX-400-4000 Hz spectrometers with tetramethylsilane (TMS) as an internal standard and DMSO-d₆, CDCl₃ as a solvent (Chemical shifts in ppm). LC-MS were recorded by Waters Micro mass Q-TOF premier Mass Spectrometer.

EXPERIMENTAL:**General procedure for the synthesis of 1-o-tolyl-3-o-tolyl-2,6-dithio-4-amino-[(2-o-tolylthiocarbamido)-1,3-benzothiazolo]-1,3,5-triazines (IIa-e)**

A reaction mixture of 2-o-tolyl-imino-6-o-tolyl imino-4-[(2-o-tolylthiocarbamido)-1,3-benzothiazolo]-1,3,5-dithiazines (Ia-e) and 10% aqueous sodium bicarbonate solution was refluxed on water bath in ethanol medium for 10 minutes. After distillation off excess solvent, brown colour crystals of 1-o-tolyl-3-o-tolyl-2,6-dithio-4-amino-[(2-o-tolylthiocarbamido)-1,3-benzothiazolo]-1,3,5-triazines (IIa-e) washed several times with petroleum ether to afford brown colour crystals of products, recrystallized from glacial acetic acid.



2-Substituted-6-o-tolylimino-4-amino-[(2-o-tolylthiocarbamido)-1,3-benzothiazolo]-1,3,5-dithiazine



1-o-tolyl-3-substituted-2,6-thio-4-amino-[(2-o-tolylthiocarbamido)-1,3-benzothiazolo]-1,3,5-triazine

where, R: -phenyl,-ethyl,-*t*-butyl, *p*-chlorophenyl, -*o*-tolyl

Properties of (IIa): It is light brown crystalline solid, melting point 252°C. Soluble in dioxane, ethanol, DMSO while insoluble in carbon tetrachloride, benzene, water, petroleum ether. Formed picrate, melting point 187°C. R_f values was found to be 0.39, for dioxane as solvent on silica gel-G with layer thickness of 0.3 mm.

RESULTS AND DISCUSSION:

Synthesis

1-o-tolyl-3-o-tolyl-2,6-dithio-4-amino-[(2-o-tolylthiocarbamido)-1,3-benzothiazolo]-1,3,5-triazine (IIa): Light Brown crystalline solid, molecular formula $C_{32}H_{27}N_7S_4$, yield 90%, melting point 252°C.; % Composition found (calculated): C-55.60 (56.51), H-3.20 (4.23), N-15.38 (15.38) and S-19.21 (20.09); FTIR [KBr, ν cm^{-1}]: 3444s, 2974s, 1678s, 1512 s, 1409 s, 1481s, 1244 s, 1138s; 1H NMR [$CDCl_3$ - $DMSO-d_6$] δ ppm]: 8.406, 2.816, 2.710, 7.001, 6.892, 6.568, 1.420, 1.378; ^{13}C ($CDCl_3$ - $DMSO-d_6$): 189.67, 160.22, 112.55, 41.82, 38.64; LC- MS : 642 .

Similarly, 2-ethylimino-6-o-tolylimino-4-amino[(2-o-tolylthiocarbamido)-1,3-benzothiazolo]-1,3,5-dithiazine (Ib), 2-*t*-butylimino-6-o-tolylimino-4-amino-[(2-o-tolylthiocarbamido)-1,3-benzothiazolo]-1,3,5-dithiazine (Ic), 2-*p*-chlorophenylimino-6-o-tolyl-imino-4-amino-[(2-o-tolylthiocarbamido)-1,3-benzothiazolo]-1,3,5-dithiazine (Id), 2-phenylimino-6-o-tolyl-imino-4-amino-[(2-o-tolylthiocarbamido)-1,3-benzothiazolo]-1,3,5-dithiazine (Ie) on isomerisation of 10% aqueous sodium bicarbonate in ethanol as a solvent by above mentioned method to isolate 1-o-tolyl-3-ethyl-2,6-dithio-4-amino-[(2-o-tolylthiocarbamido)-1,3-benzothiazolo]-1,3,5-triazine (IIb), 1-o-tolyl-3-*t*-butyl-2,6-dithio-4-amino-[(2-o-tolylthiocarbamido)-1,3-benzothiazolo]-1,3,5-triazine (IIc), 1-o-tolyl-3-*p*-chlorophenyl-2,6-dithio-4-amino-[(2-o-tolylthiocarbamido)-1,3-benzothiazolo]-1,3,5-triazine (IId), 1-o-tolyl-3-phenyl-2,6-dithio-4-amino-[(2-o-tolylthiocarbamido)-1,3-benzothiazolo]-1,3,5-triazine (IIe) respectively by above mentioned method and enlisted in **Table No.I**

Table No. I

Sr. No.	Compd. No	1-o-Tolyl-3-Substituted-2,6-dithio-4-amino-[(2-o-tolylthiocarbamido)-1,3-benzothiazolo]-1,3,5-triazine (IIb-IIe)	Yield (%)	m.p (°C)
1	[IIb]	1- <i>o</i> -Tolyl-3-Ethyl-2,6-[(2- <i>o</i> -Tolylthiocarbamido)-1,3-benzothiazolo]-1,3,5-triazine	85	230
2	[IIc]	1- <i>o</i> -Tolyl-3- <i>t</i> -Butyl-2,6-[(2- <i>o</i> -Tolylthiocarbamido)-1,3-benzothiazolo]-1,3,5-triazine	79	196
3	[IId]	1- <i>o</i> -Tolyl-3- <i>p</i> -Chlorophenyl-2,6-[(2- <i>o</i> -Tolylthiocarbamido)-1,3-benzothiazolo]-1,3,5-triazine	73	215
4	[IIe]	1- <i>o</i> -Tolyl-3-Phenyl-2,6-[(2- <i>o</i> -Tolylthiocarbamido)-1,3-benzothiazolo]-1,3,5-triazine	80	200

CONCLUSION:

In the current study focused on the synthetic route of 1-o-tolyl-3-substituted-2,6-dithio-4-amino-[(2-o-tolylthiocarbamido)-1,3-benzothiazolo]-1,3,5-triazine s. This method is cheaper and convenient and less time consumable.

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