



CODEN [USA]: IAJPBB

ISSN: 2349-7750

**INDO AMERICAN JOURNAL OF
PHARMACEUTICAL SCIENCES**<http://doi.org/10.5281/zenodo.1186144>Available online at: <http://www.iajps.com>

Research Article

SYNTHESIS OF 1-SUBSTITUTED-2-THIO-4-AMINO-6-PHENYLFORMAMIDINO, 1, 3, 5-TRIAZINES

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Amravati 444606.**Abstract:**

Recently in this laboratory 1-substituted-2-thio-4-amino-6-phenylformamidino 1,3,5-triazines (XIIIa-g) were synthesised by isomerisation of 2-substituted imino-4-amino-6-formamidino-1,3,5-thiadiazines (XIIa-g) successfully by refluxing with 10% aqueous ethanolic sodium bicarbonate medium. The structure of all the synthesized compounds was justified on the basis of chemical characteristics, elemental analysis and spectral studies.

Keywords: 10% aqueous ethanolic, sodium bicarbonate.**Corresponding author:****D.T.Tayade,**Department of Chemistry,
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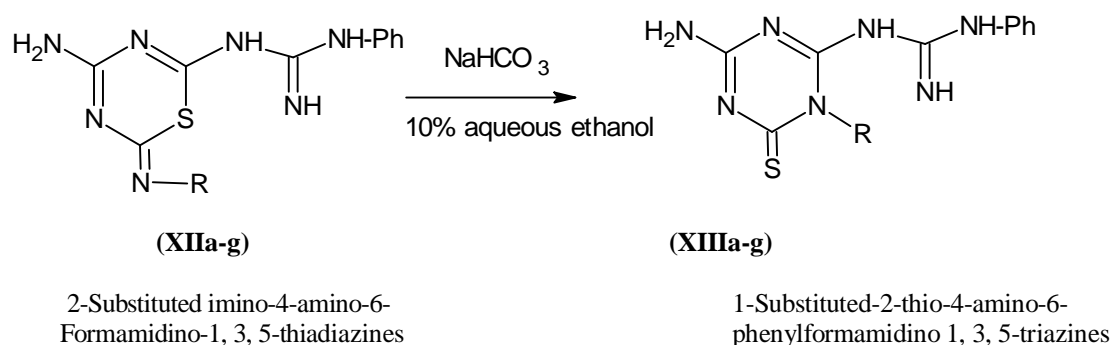


Please cite this article in press as D.T.Tayade* and S.P. Ingole, *Synthesis of 1-Substituted-2-Thio-4-Amino-6-Phenylformamidino, 1, 3, 5-Triazines*, Indo Am. J. P. Sci, 2018; 05(02).

INTRODUCTION:

The 1,3,5-triazine nucleus containing compounds having huge importance in human life due to their varieties of applications in medicinal, industrial pharmaceutical and agricultural fields [1-6]. These 1,3,5-triazines have their own identity and importance in medicinal [7], pharmaceutical [8], agricultural [9] and industrial [10] fields few of them possesses antidiabetic [11,12], anti-tumor [13-16], anti-inflammatory [17], anti-depressant [18], hypoglycaemic [19] activities. They are also used as herbicidal [20-26], fungicidal [27-29],

Insecticidal [30], anti-corrosive [31], antimicrobial [32] and anti-convulsant properties. Hence it was thought interesting to carry out the isomerisation of 2-substitutedguanidino-4-substitutedimino-6-substitutedimino-1, 3, 5-thiadiazines (**XIIa-g**) into 1-substituted-2-substitutedguanidino-4-substitutedimino-6-thio-1, 3, 5-triazines (**XIIIa-g**) in the presence of 10% ethanolic sodium bicarbonate medium. The tentative reaction for the formation of product is depicted below (**Scheme-I**).

**Scheme-I****General remarks**

All reagents were purchased from commercial suppliers and used without further purification. Dry methanol and diethyl ether were purchased from Aldrich and were used as such. All reactions were run in oven-dried round bottom flask or vial containing a teflon-coated stir bar and sealed with septum. Analytical thin layer chromatography was carried out on silica pre-coated glass plates (Silica gel 60 F254, 0.25 mm thickness) and visualized with UV light at 254 nm. ^1H NMR spectra were recorded on Bruker 400-MHz Ultrashield Advance II 400 model (400 and 100 MHz, respectively) at ambient temperature with CDCl_3 or DMSO-d_6 as solvents. Data for ^1H are recorded as follows: δ chemical shift (ppm), multiplicity (s, singlet; d, doublet; dd, double doublet; t, triplet; q, quartet; m, multiplet), coupling constant (Hz), integration. Spectra were referenced internally to the residual proton resonance in CDCl_3 (δ 7.26 ppm), DMSO-d_6 (δ 2.50 ppm) or with tetramethylsilane (TMS, δ 0.00 ppm) as the internal standard. Chemical shifts (δ) were reported as part per million (ppm) in δ scale downfield from TMS.

RESULT AND DISCUSSION:**General procedure for the Synthesis of 1-ethyl-2-formamidino-4-amino-6-thio-1,3,5-triazine (XIIIa)**

2-Ethylimino-4-amino-6-formamidino-1,3,5-thiadiazine (**XIIa**) was suspended in 10% ethanolic sodium bicarbonate solution and refluxed for half an hour on water bath. During heating the reactant went into the solvent. After distillation of excess solvent milky white crystals were isolated and recrystallised from glacial acetic acid to obtain (**XIIIa**), Yield 78%, m.p. 185°C .

Properties of [XIIIa]

It is brown colour crystalline solid having melting point 185°C . It gave positive test for nitrogen and sulphur. It was desulphurized by alkaline plumbite solution which clearly indicate the presence of $\text{C}=\text{S}$ group. It was soluble in water, ethanol, DMSO-d_6 while insoluble in carbon tetrachloride, chloroform, benzene, petroleum ether. It formed picrate having melting point 209°C . **Elemental analysis:** [C: 61.20% (found), 62.10% (calculated)], [H: 03.51% (found), 04.99 % (calculated)], [N: 18.11% (found), 18.11 % (calculated)], [S: 10.84% (found), 11.82 % (calculated)]. **IR Spectrum:** The IR spectrum was carried out in KBr-pellets. The important absorptions are correlated as (cm^{-1}) 3180.62 N-H stretching, 2895.15 C-H stretching, 1726.89 N=C-N stretching, 1514.12 N-C=S stretching, 1288.45 C-N stretching,

1010.70 C=S stretching. **NMR Spectrum:** The NMR spectrum was carried out in DMSO-d₆ and CDCl₃. This spectrum distinctly displayed the signals due to Ar-H protons at δ 8.4227-6.9756 ppm, -NH proton at δ 3.6302-3.0104 ppm, -CH₃ protons at δ 1.3791-1.3437 ppm.

Similarly, 2-phenylimino-4-amino-6-phenylformamidino-1,3,5-thiadiazine (**XIib**), 2-methylimino-4-amino-6-phenylformamidino-1,3,5-thiadiazine (**XIic**), 2-p-chlorophenylimino-4-amino-6-phenylformamidino-1,3,5-thiadiazine (**XIid**), 2-o-tolylimino-4-amino-6-phenylformamidino-1,3,5-thiadiazine (**XIie**), 2-m-tolylimino-4-amino-6-

phenylformamidino-1,3,5-thiadiazine (**XIif**) and 2-p-tolylimino-4-amino-6-phenylformamidino-1,3,5-thiadiazine (**XIig**) were isomerised by 10% aqueous ethanol to isolate 1-phenyl-2-thio-4-amino-6-phenylformamidino-1,3,5-triazine (**XIIIb**), 1-methyl-2-thio-4-amino-6-phenylformamidino-1,3,5-triazine (**XIIIc**), 1-p-chlorophenyl-2-thio-4-amino-6-phenylformamidino-1,3,5-triazine (**XIII d**), 1-o-tolyl-2-thio-4-amino-6-phenylformamidino-1,3,5-triazine (**XIIIe**), 1-m-tolyl-2-thio-4-amino-6-phenylformamidino-1,3,5-triazine (**XIII f**), 1-p-tolyl-2-thio-4-amino-6-phenylformamidino-1,3,5-triazine (**XIIIg**) and enlisted in **Table-1**

Table-1

Sr. No.	1-substituted-2-thio-4-amino-6-phenylformamidino-1,3,5-triazines	Yield %	M. P.
1.	1-methyl-2-formamidino-4-amino-6-thio-1,3,5-triazine	94	272
2.	1-methyl-2-formamidino-4-amino-6-thio-1,3,5-triazine	94	272
3.	1-p-chlorophenyl-2-formamidino-4-amino-6-thio-1,3,5-triazine	92	275
4.	1-o-tolyl-2-formamidino-4-amino-6-thio-1,3,5-triazine	98	222
5.	1-m-tolyl-2-formamidino-4-amino-6-thio-1,3,5-triazine	94	247
6.	1-p-tolyl-2-formamidino-4-amino-6-thio-1,3,5-triazine	94	268

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