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Research Article

**SYNTHESIS OF 4-(2-THIO-4-SUBSTITUTEDIMINO-1,3,5-
TRIAZ-6-YL)-PYRIDINES (IIIa-d)**S.G. Khobragade¹, D.T. Tayade², N.S.Dixit³

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Abstract:

A new series of 4-(2-thio-4-substitutedimino-1,3,5-triaz-6-yl)-pyridines (IIIa-d) was successfully synthesized by the interactions of 1-(4-pyridineimino)thiocarbamide (I) with various isocyanodichlorides (IIa-d) in acetone medium. The synthesized compounds were recrystallized and their structure was justified and established on the basis of elemental analysis, chemical characteristics and through spectral studies.

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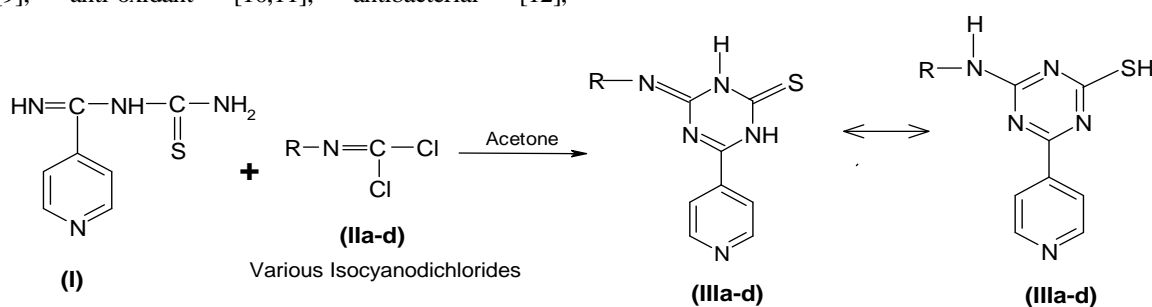
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INTRODUCTION:

The literature survey reveals that the heterocyclic compounds containing nitrogen and nitrogen and sulphur in the ring had gained immense importance in human life due to their noticeable and varieties of applications in pharmaceutical [1,2], medicinal [3], industrial [4] and agricultural sciences[5]. The heterocycles containing s-triazines in the nucleus had been successfully tested against various microbes and it was found that they possess potential therapeutical values [6] for several diseases. So these compounds possess their own importance in medical faculty, pharmaceutical, industrial and agricultural fields. Some triazines contain anticancer [7], antitumor [8], antidiabetic [9], anti-oxidant [10,11], antibacterial [12],

anticonvulsant [13], antiinflammatory [14], blood pressure deprent [15], hypoglycaemic [16], muscle relaxant [17] properties. Some of them are used in industries such as herbicidal [18-20], sea water algeacidal[21], fungicidal [22] and insecticidal [23]. Some triazino compounds also showed remarkable antimicrobial activities [24,25].

Up till now the synthesis of 4-(2-thio-4-substitutedimino-1,3,5-triaz-6-yl)pyridine (**IIIa-d**) was not carried out. Thus 4-(2-thio-4-substitutedimino-1,3,5-triaz-6-yl)pyridines (**IIIa-d**) were successfully synthesized by the interaction of 1-(4-pyridineimino)thiocarbamide (**I**) with various isocyanodichlorides (**IIa-d**) in acetone medium.



1-(4-Pyridineimino)thiocarbamide

4-(2-Thio-4-substitutedimino-1,3,5-triaz-6-yl)pyridines

Where, R = – phenyl, –methyl, –ethyl, –t-butyl,

EXPERIMENTAL:

The melting points of all the synthesized compounds were recorded using hot paraffin bath and are uncorrected. The carbon and hydrogen analysis was carried out on Carlo-Ebra-1106 analyser, nitrogen estimation was carried out on Colman-N-analyser-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 AC-300F spectrometer with TMS as internal standard using CDCl_3 and DMSO-d_6 as solvent. The purity of the compounds was checked on Silica Gel-G plates by TLC with layer thickness of 0.3 mm. All chemicals used were of AR grade (Indian make) except allylthiourea Lancaster (Germany make). Alkyl/Aryl isothiocyanates, isothiocarbamoylchloride, isocyanodichlorides and phenylthiourea have been prepared by known literature methods.

RESULT AND DISCUSSION:**Synthesis of 4-(2-thio-4-phenylimino-1,3,5-triaz-6-yl)pyridine (IIIa):****Scheme I**

4-(2-Thio-4-phenylimino-1,3,5-triaz-6-yl)pyridine (**IIIa**) was synthesized by refluxing the mixture of 1-(4-pyridineimino)thiocarbamide (**I**) with phenylisocyanodichloride (**IIa**) in acetone medium for 4 hours, during boiling the suspended (**I**) and (**IIa**) went into the solution and new products was found to be gradually separated out. It was filtered in hot conditions and then on basification with dilute ammonium hydroxide solution it gave yellow crystalline product. Dried at room conditions and recrystallized with ethanol, yield 89%, m.p. 148°C.

Properties: It is dark yellow crystalline solid having melting point 148°C. It gave positive test for nitrogen and sulphur (negative test for chloride which clearly indicated removal of chlorine). It gave yellow precipitated in alkaline plumbite test which clearly indicate –SH group is present. The benzene solution of compounds when treated with pure and dry carbon disulphide then colourless solution was obtained indicating =NH (imino) group is not present²⁶. It formed picrate having melting point 165°C. It was soluble in water, ethanol, DMSO-d_6 , benzene, dioxane and petroleum ether and insoluble in carbon tetrachloride, chloroform. **Elemental**

Analysis: C [(found 59.36%) calculated 59.78%], H [(found 03.48%) calculated 03.91%], N [(found 24.83%) calculated 24.91%], S [(found 11.38%) calculated 11.38%]. **IR Spectra:**-The IR spectra was carried out in KBr pellets and the important absorption can be correlated as (cm⁻¹) 3366.51 (NH stretching), 3010.50 (Ar-H stretching), 2660.50 (S-H stretching), 1589.47 (N=C=N grouping showing Hexocyclic ring), 1466.53 [C=N stretching (Ring)], 1093.58 (C-N stretching), 724.61 (Monosubstituted Benzene), 619.61 (C-S stretching). **NMR Spectra:**-The spectrum was carried out in CDCl₃ and DMSO-d₆. This spectrum distinctly displayed the signals due to pyridino proton at δ 8.0136-

8.9814 ppm, Ar-H proton at δ 7.0263-7.9980 ppm, SH proton at δ 5.7331-5.9901 ppm, NH proton at δ 5.3007-5.3216 ppm.

Similarly, 4-(2-thio-4-methylimino-1,3,5-triaz-6-yl)pyridine (**IIIb**), 4-(2-Thio-4-ethylimino-1,3,5-triaz-6-yl)pyridine (**IIIc**) 4-(2-thio-4-t-butylimino-1,3,5-triaz-6-yl)pyridine (**III d**) were synthesized by the interactions of methylisocyanodichloride (**IIb**), ethylisocyanodichloride (**IIc**) t-butylisocyanodichloride (**IId**) with 1-(4-pyridineimino)thiocarbamide (**I**) in acetone medium respectively by the above mentioned method **Experiment No. 2 to 4** and enlisted in **Table No. I**

Table No. I

Sr. No.	Expt. No.	4-(2-Thio-4-substitutedimino-1,3,5-triaz-6-yl)pyridines (IIIa-h)	Yield (%)	M.P. °C
1.	2methyl.....	90	138
2.	3ethyl.....	84	142
3	4t-butyl.....	84	149

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