



**SYNTHESIS AND ANALYSIS OF NOVEL 1,3,5-TRIAZINES BY SELECTIVE
DETERBUTYLATION OF 3- ARYL/ALKYL – 4 -T- BUTYLIMINO – 5-T-BUTYL -
6-T BUTYL MERCAPTO – TETRAHYDRO- 1,3,5 – TRIAZINES.**

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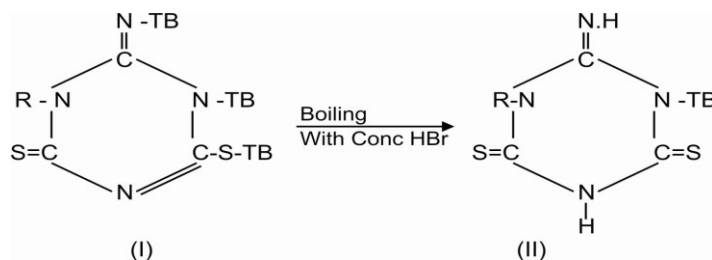
ABSTRACT

The deterbutylation of 3 – aryl/alkyl – 4 – t- butyl imino – 5 – t butyl – 6 tbutyl mercapto – tetra hydro- 1,3, 5 – triazines (I) has been carried out with the help of boiling conc. Hydrobromic acid. Due to selective deterbutylation, the t-butyl group attached to sulphur at position 6 and t-butyl group attached to imino nitrogen at position 4 cleaved off and a new product 3 – aryl/ alkyl – 4- imino – 5 – t butyl – 2,6, dithiohexahydro – 1,3,5 triazines (II) has been isolated. The characterization of the compounds have been carried out by the usual chemical transformation and spectral data.

Keywords: 1,3,5- triazines, IR Spectrum, ^1H NMR studies.

INTRODUCTION:

Applications of t – butyl group as a blocking group in the synthesis of Nitrogen and Sulphur compounds have been investigated earlier. In view of our interest in the Synthesis of several 1,3,5- triazines and their derivatives. Several 1,3,5, triazines containing S-t- butyl and N – t – butyl groups have been synthesized by the interaction of 1 – t butyl – 2-s-t-butyl-5-aryl – 2, 4 – isodithiobiurets and N-t-butyl – isocyanodichloride. The corresponding 1,3,5- triazines on boiling with conc. Hydrobromic – acid produce varieties of 1,3,5 – triazines. These compounds are important class of organic compounds.





Experimental:

Chemical: Analytical Reagent Grade Chemicals were used.

1) Preparation of 2- S- t – butyl – 1 – t butyl- 5 aryl/alkyl 2,4 – dithiobiurets ^{6,7}.
These have been prepared by the interaction of 2-S-t-butyl-1-tbutyl thiocarbamide with aryl/alkyl isothiocyanate^{6,7}.

2) Preparation of 3-Aryl/alkyl-4-t-butyl-imino-5 -t-butyl-6-tbutyl mercapto-tetrahydro-1,3,5 – triazines (I) ^{9,11}.


3) Preparation of 3-Aryl/alkyl-4-imino-5t-butyl-2,6,dithiohexahydro-1,3,5, triazines (II) (Where aryl=Phenyl).

3- Phenyl – 4-t-butylimino-5-tbutyl-6-t-butyl- mercapto-tetrahydro-1,3,5-triazine Ia (2.0g) was mixed with conc. Hydrobromic acid (48%,20ml) and the reaction mixture was refluxed over a gentle flame for 2 hr. The solid gradually went into solution. The reaction mixture on cooling and treatment with dilute ammonium hydroxide afforded a white solid (1.5g) IIa. It was crystallized from ethanol; m.p. 137^o (Found :N: 18.85%,S:21:3% C₁₃H₁₆N₄S₂ requires N:19.18%,21.92%).

The product with m.p. 137^o was found insoluble in hot acetone and benzene. It was found non-desulphurisable when boiled with alkaline plumbite solution. On pyrolysis, smells of t-butylisothiocyanate and phenylisothiocyanate were quite perceptible.

RESULT AND DISCUSSION :

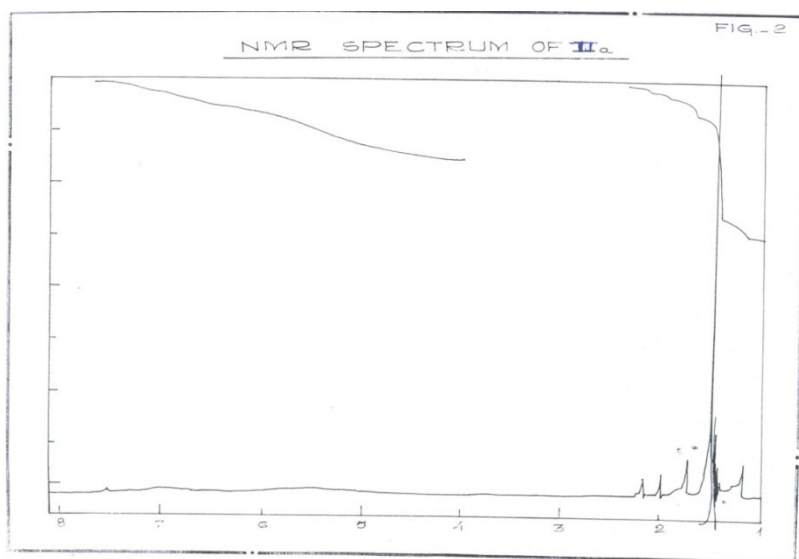
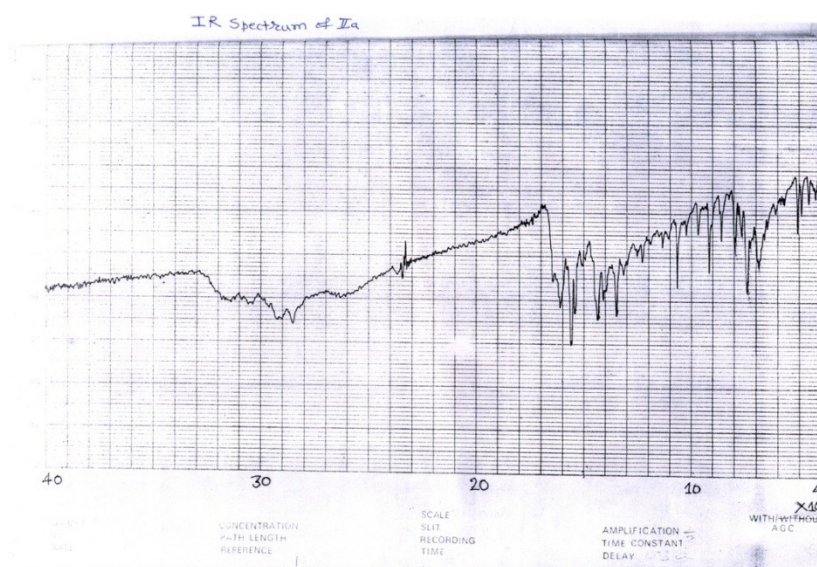
The main absorption bands observed in the IR spectrum of (IIa (Fig. I) are given below :

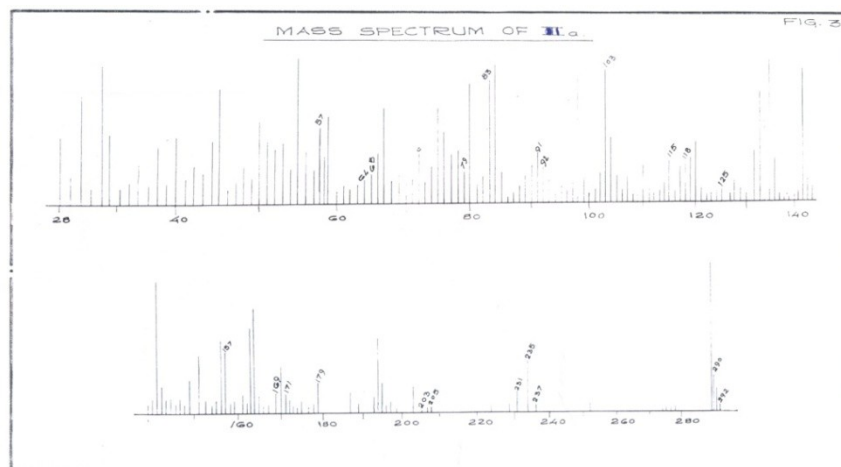
Absorption observed (cm ⁻¹)	Assignment	Absorption expected (cm ⁻¹)
3330	-NH- stretching	3450-3125 ^{6a,1b}
1640	N-C=N grouping showing the presence of exocyclic imino group	1685-1582 ^{7a}
1584	1,3,5- triazine quadrant	1600-1500 ^{7b,8b}
1230	 C=S grouping	1400-700 ^{8c}
1220	Tert – butyl group	1255-1210 ^{7a}
790	Isoform of the 1,3,5-triazine ring	795-750 ^{7b,1c}
700	Five adjacent hydrogen	710-690 ^{8b}



The NMR of the product IIa (Fig. 2) showed peaks due to methyl protons of butyl α 1.4 at ppm and aromatic protons at ppm and aromatic protons at 7.6 ppm^{7,8}. A signal at α 2.1 ppm indicated the NH proton.

The mass spectrum of the product was also recorded (Fig. 3). The molecular ion peak was located in this spectrum while other important peaks were also recorded. The probable fragmentation patterns of the molecular ion are shown in Scheme 1.





Scheme - 1

Mass Spectral data of IIa

M+[C ₁₃ H ₁₆ N ₄ S ₂] ⁺	292
[C ₉ H ₇ N ₄ S ₂] ⁺	235
Protonated [C ₉ H ₇ N ₄ S ₂] ⁺	237
[C ₇ H ₁₁ N ₄ S ₂] ⁺	215
Protonated [C ₇ H ₁₁ N ₄ S ₂] ⁺	217
[C ₉ H ₇ N ₄ S] ⁺	203
Protonated [C ₉ H ₇ N ₄ S] ⁺	205
[C ₇ H ₁₁ N ₄ S] ⁺	183
Protonated [C ₇ H ₁₁ N ₄ S] ⁺	185
C ₈ H ₇ N ₃ S] ⁺	177
C ₆ H ₄ N ₃ S] ⁺	157
C ₆ H ₁₂ N ₃ S] ⁺	158
C ₇ H ₅ NS] ⁺	135
C ₅ H ₉ NS] ⁺	115
C ₇ H ₅ N] ⁺	103
C ₅ H ₉ N] ⁺	83
C ₆ H ₅] ⁺	77
C ₆ H ₄] ⁺	76
C ₄ H ₉] ⁺	57



Table – 1

3-Aryl/alkyl-4-imino-5-t-butyl-2,6-dithiohexahydro-1,3,5- triazines (II)

S. No.	3-Aryl/alkyl-4-imino-5-t-butyl-2,6-dithiohexahydro-1,3,5-triazine (II)		Yield, G	M.P. °C
1	3- phenyl -	Ila	1.5	137
2	3-o - tolyl -	Ilb	1.2	143
3	3-p-tolyl-	Ilc	1.5	140
4	3-p-chloro-phenyl-	Ild	1.3	175

CONCLUSION

These compounds are important class of organic compounds. Triazines have wide applications in different industries such as Textile industries & Medicinal Chemistry. Nitrogen and Sulphur containing ring compounds have diverse chemical reactivity.

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