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Synthesis of 1-3 Thiazines from Aurone

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ABSTRACT

A large group of dyes has the phenothiazine structure, including methylene blue thiazine are use for dyes, tranquilizers and insecticides thiazine can help reduce some of that extra water weight you may be holding on to in your stomach.thiazine is a fairly basic diuretic supplement, it reduce water and increase vascularity, so it is also use as anabolic agent in medicine. It is therefore reported that thiourea reacts with mesityl oxide in acetic acid medium to give 2-imino-6H, 2,3dihydro-1-3-thiazine and 2-thioxotetrahydro pyridine derivatives.

Key words: 1-3 Thiazines from Aurone, Hetrocyclic compounds.

INTRODUCTION

Six members heterocyclic compound containing one nitrogen and one sulphur atom are known as thiazines.Chalcone condence with thiourea to give 1,-3 thiazine¹. According to Halr and Brill²-. condensation of 5-methyl thiouronium sulphate with nitromalonic dialdehyde result in the formation of 2- amino -5-nitro-1,3,2-thiazine. However Boarland and Mcomie³ obtained only 5nitro -5-(1-piperidine) and unable to confirm this report.

Many compounds of thiazines are known, phenothiazine has been use as vermifuge for livestock and also as an insecticide.drugs of the phenothizine type include chlorpromazine, a tranquillizer, promethazine hydrochloride (phenergan), a long-acting antihistaminic; diethazine hydrochloride (Diparcol), used in treatment of Parkinson's.

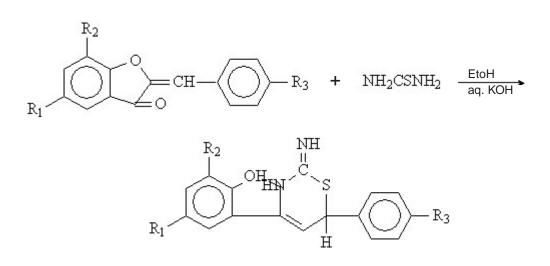
A large group of dyes has the phenothiazine structure, including methylene blue thiazine are use for dyes, tranquilizers and insecticides thiazine can help reduce some of that extra water weight you may be holding on to in your stomach.thiazine is a fairly basic diuretic supplement, it reduce water and increase vascularity, so it is also use as anabolic agent in medicine. It is therefore reported that thiourea reacts with mesityl oxide in acetic acid medium to give 2imino-6H, 2,3dihydro-1-3-thiazine and 2thioxotetrahydro pyridine derivatives⁴ similarly 6hydroxy -4-methoxy-5-aryl-acryloyl benzo (b) furanon reaction with thiourea in alkaline medium afforded 6- aryl -4(5(6-hydooxy-4, methoxy benzo (B) furanyl)-2-imino-6-H-2,3-dihydro-1,3-thiazines.⁵.

4-6diaryl2-imino6-H-2, 3dihydro1,3 thiazines. Were prepared by chincholkar, kakade, Jamode6 from 2-hydroxy chalcone with thiourea in alkaline medium. Recently 1,3 thiazine were prepared from b-(2,furyl) acrylophenones⁷ and from nitrochalcones⁸.No such work is referred in literature for the preparation 1-3 thiazines from aurone therefore it is thought of interest to study the preparation of 1-3 thiazines from aurone. Aurone on treatment with thiourea in presence of aq KOH and ethanol gave 4.6-diaryl-2imino-6-H-2,3dihydro-1,3 thiazines

Detail of preparation

Aurone (0.01 mole) and thiourea (0.01) were dissolved in ethanol (30 ml). To this solution aq KOH solution(0.02 mole) was added. The reaction mixture was refluxed for 3 hours and diluted with water, acidified with 1:1 HCL, A brownish solid thus separated was filtered and crystallized from ethanol using the same procedure, the other 1,3-thizines were prepared.

S. No	Aurone -2-Imino-6H-2,3-dihydro-1,3 thiazines		
1.	2-(4' methoxy benzylidene)- 5-methyl- coumaranone	4-(2-hydroxy-5-methyl phenyl)-6- (-4-methoxyphenyl-	246
2.	2-benzylidene –5-methyl coumarone	4-(2-hydroxy-5-methyl phenyl-6-phenyl-	216
3.	2-(4' methoxy benzylidene)-3-bromo 5-methyl coumaranone	4-(2-hydroxy-3bromo-5methyl phenyl)-6- (-4-methoxy phenyl)-	230
4.	2-benzylidene-3bromo-5-methyl	4 -(2-hydroxy-3bromo-5methyl phenyl)-6 phenyl -coumaranone	140
5.	2-(4' methoxy benzylidene)-3-nitro 5-methyl coumaronone	4 -(2-hydroxy-3nitro-5methyl phenyl)- 4-methoxy phenyl-	232
5.	2-benzylidene –3-nitro-5-methyl coumaranone	4 -(2-hydroxy-3nitro-5methyl phenyl)- 6(phenyl)-	210



Properties of Product A

- It gives coloration with ferric chloride solution, which indicate presence of free phenolic OH group.
- 2) It gives deep blue coloration with conc. H_2SO_4 showing the absence of -C-CH=CH- linkage.
- From elemental analysis, the molecular formula of the compound is C₁₈H₁₈O₂N₂S
- C: found 63.57%
- H: found5.35%
- N: found 9.27%
- S: found 10.59%
- C: calculated 63.32% H: calculated 5.21%
- N calculated 9.12%
- S: calculated10.14%

IR

IR spectrum was recorded in nujol and reproduced on plate B

Region	Frequency	Co-relation	
3700-2800	2940	strongly H bonded stretching	
3000 2800	2940	C-H streching	
1700-1600	1620	C=N streching	
1650-1500	1585	N-H streching	
1300-900	1268	C-N streching	
1260-1050	1187	Ar-o streching in aromatic ether	

The observed chemical shift can be co related as follows

Chemical shift	Nature of peak	No. of proton	Type of proton
2.5	S	3H	Ar-CH3
3.32	d	1H	-C-CH-S
3.9 🖌	S	ЗH	AR-OCH3
5.24 🖍	S	1H	-C=NH
6.52-7.33 よ	m	7H	Ar-H
8.03 🖌	br	2H	N-H
11.5 🖌	S	1H	Ar-OH

NMR spectrum

 ${\rm PMR}\,$ spectrum was recorded ~ in ${\rm CDCI}_{\rm 3}$ with TMS as an internal standard and is reproduced on plate c.

Hence the compound is 4 (2- hydroxyl -5methyl phenyl) 6- (4-methoxy phenyl)-2 imino -6 H -2,3-dihydro-1,3 thiazine

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