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2-[(2-THIO-3-PHENYL-6-SUBSTITUTEDAMINO)-1,3,5-THIDIAZINO]IMINO-11-(PIPRAZINE-1-YL)DIBENZO[b,f][1,4]OXAZEPINES

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Keywords: 2-[(2-substitutedimino-6-substitutedamino)-1,3,5-dithiozino]imino11-(pipr- azine-1-yl)dibenzo[b,f][1,4] oxazepines, 5% aqueous sodium bicarbonate, ethanol

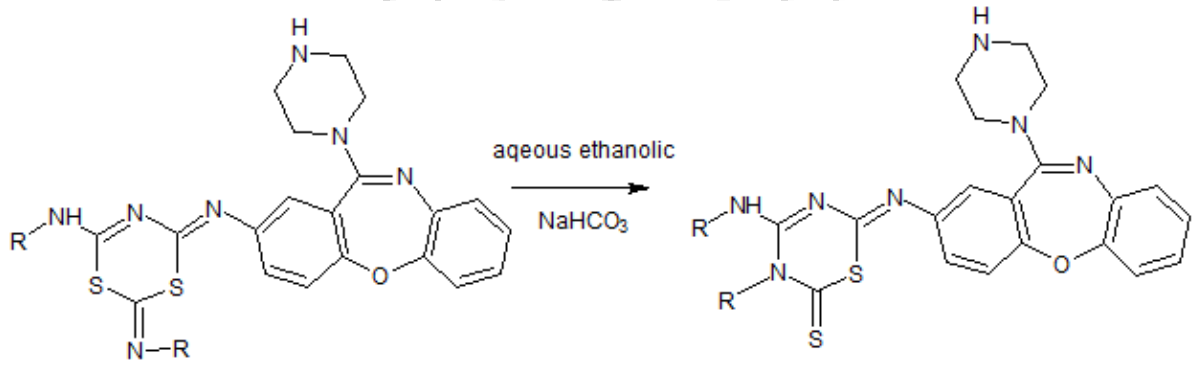
ABSTRACT

Recently a novel series of 2-[(2-thio-3-substituted-6-substitutedamino)-1,3,5-thidiazino] imino11-(piprazine-1-yl)dibenzo[b,f][1,4] oxazepines **[IXB(a-f)]** was successfully synthesized by the isomerisation of 2-[(2-substitutedimino-6-substitutedamino)-1,3,5-dithiozino] imino 11-(piprazine-1-yl) dibenzo [b,f][1,4] oxazepines **[VIII(a-f)]** by 5% aqueous sodium bicarbonate in ethanol medium. The structures of all synthesized compounds were justified on the basis of chemical characteristics, elemental analysis and spectral studies

INTRODUCTION

Oxazepine and their derivatives have some important biological pharmacological activities[1] such as enzyme inhibitors[2], analgesic[3], anti-depressant[4] and psychoactive drugs[5]. Oxazepine nucleus is used for treatment of depression, anxiety and agitation[6-7]. Recently new series of 1,2,4-thiadiazoles, 1,3,5-thiadiazines and 1,3,5-dithiazines were synthesized by exploring the synthetic applications of –thiocarbamido, -amino, -halo, -cyano, etc. and their antimicrobial, antifungal, antibacterial, analgesic physiochemical parameters[8-11] were studied. 2-Chloro-11-(piperazin-1-yl)dibenzo [b,f] [1,4] oxazepine **(IB)** and their derivatives showed agricultural, medicinal, biological, pharmaceutical, industrial significances and applications.

The main objective of the work is to synthesize a novel series of 2-[(2-phenylimino-6-substitutedamino)-1,3,5-dithiazino]imino-11-(piprazine-1-yl) dibenzo [b,f] [1,4] oxazepines [**VIIIB(a-f)]**. These were synthesized by the isomerisation of 2-[(2-substitutedimino-6-substitutedamino)-1,3,5-dithiozino]imino-11-(piprazine-1-yl) dibenzo [b,f][1,4] oxazepines **[VIII(a-f)]** by 5% aqueous sodium bicarbonate in ethanol, **Scheme-1.**



[VIIIB(a-f)]
2-[(2-substitutedimino-6-substitutedamino)
-1,3,5-dithiozino] imino 11-(piprazine-1-yl)
dibenzo [b,f][1,4] oxazepines

[IXB(a-f)]
2-[(2-thio-3-substituted-6-substitutedamino)
-1,3,5-thidiazino]imino11-(piprazine-1-yl)
dibenzo[b,f][1,4]oxazepines

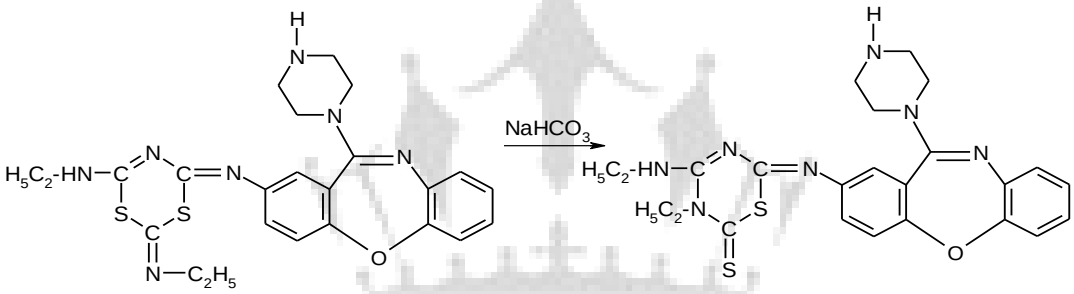
Where, R= -methyl, -ethyl, -t-butyl, -phenyl, -p-chlorophenyl, -p-tolyl.

Scheme-1

Synthesis of 2-[(2-thio-3-ethyl-6-ethylamino)-1,3,5-thidiazino]imino-11-(piprazine-1-yl)dibenzo[b,f][1,4]oxazepine

Synthesis of 2-[(2-thio-3-ethyl-6-ethylamino)-1,3,5-thidiazino]imino 11-(piprazine-1-yl)dibenzo[b,f][1,4]oxazepine **[IXB(a)]** was carried out by isomerising 2-[(2-substituted imino-6-substitutedamino)-1,3,5-dithiozino]imino-11-(piprazine-1-yl) dibenzo [b,f][1,4] oxazepine **[VIIIB(a)]** in 5% aqueous sodium bicarbonate solution in ethanol. After distillation of excess solvent yellow crystals were separated out and were recrystallized from glacial acetic acid. Yield 94 %, M.P. 240˚C.

The p formation of **[IXB(b)]** is depicted below,



[VIIIB(a)]

2-[(2-Ethylimino-6-phenylamino)-1,3,5-dithiozino]imino-11-(piperazin-1-yl) dibenzo[b,f][1,4]oxazepine

[IXB(a)]

2-[(2-Thio-3-ethyl-6-ethylamino)-1,3,5-dithiozino]imino-11-(piperazin-1-yl) dibenzo[b,f][1,4]oxazepine

[IXB(a)]

Properties of [IXB(a)]

It is brown colour crystalline solid having melting point 240^0C. It gave positive test for nitrogen and sulphur. It was desulphurized by alkaline plumbite solution which clearly indicate the presence of C=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 209^0C. **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-d_6 and $CDCl_3$. 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, -CH_3 protons at δ 1.3791-1.3437 ppm.

Similarly, 2-[(2-ethylimino-6-phenylamino)1,3,5-dithiazino]imino-11-(piperazine-1-yl) dibenzo [b,f] [1,4]oxazepine **[VIIIB(b)],** 2-[(2-ethylimino-6-methylamino)-1,3,5-dithia zino]imino-11-(piperazine-1-yl)dibenzo [b,f] [1,4] oxazepine **[VIIIB(c)],** 2-[(2-ethylimino-6-tertbutylamino)-1,3,5-dithiazino] imino-11-(piperazine-1-yl)dibenzo [b,f] [1,4]oxazepine **[VIIIB(d)],** 2-[(2-ethylimino-6-p-chlorophenylamino)-1,3,5-dithiazino] imino-11-(piper azine-1-yl)dibenzo[b,f][1,4]oxazepine**[VIIIB(e)],** 2-[(2-ethylimino-6-p-tolylamino)-1,3,5-dithiazino]imino-11-(piperazine-1-yl)dibenzo[b,f][1,4]oxazepine **[VIIIB(f)]** were isomerized by 5% aqueous sodium bicarbonate solution by above mentioned method to isolate 2-[(2-thio-3-ethyl-6-phenylamino)-1,3,5-thiadiazino] imino-11-(piperazine-1-yl)dibenzo[b,f][1,4] oxazepine **[IXB(b)],** 2-[(2-thio-3-ethyl-6-methylamino)-1,3,5-thiadiazino]imino-11-(piper azine-1-yl)dibenzo [b,f][1,4] oxazepine **[IXB(c)],** 2-[(2-thio-3-ethyl-6-tertbutylamino)-1,3,5-thiadiazino]imino-11-(piperazine-1-yl)dibenzo[b,f][1,4]oxazepine **[IXB(d)],** 2-[(2-thio-3-ethyl-6-p-chorophenylamino)-1,3,5-thiadiazino]imino-11-(piperazine-1-yl)dibenzo [b,f] [1,4] oxazepine **[IXB(e)],** 2-[(2-thio-3-ethyl-6-p-tolylamino)-1,3,5-thiadiazino]imino-11-(piperazine-1-yl)dibenzo[b,f][1,4] oxazepine **[IXB(f)],** by the above mentioned method and enlisted in **Table No. I**

Table No. I

Sr. No.	**Compd. No.**	2-[(2-ethylimino-6-**substituted**amino)1,3,5-dithiazino]imino-11-(piperazine-1-yl) dibenzo [b,f] [1,4]oxazepine	**Yield (%)**	**M.P. (^{0}C)**
1	**[IXB(b)]**	2-[(2-Thio-3-ethyl-6-**phenyl**amino)------- oxazepine	88	164
2	**[IXB(c)]**	2-[(2-Thio-3-ethyl-6-**methyl**amino)------- oxazepine	92	148
3	**[IXB(d)]**	2-[(2-Thio-3-ethyl-6-**t-butyl**amino)------ oxazepine	89	184
4	**[IXB(e)]**	2-[(2-Thio-3-ethyl-6-**p-chloro**phenyl amino) ------- oxazepine	86	190
5	**[IXB(f)]**	2-[(2-Thio-3-ethyl-6-**p-tolyl**amino)------ oxazepine	90	197

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