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SYNTHESIS OF 2-[(2-PHENYLDIMINO-6-SUBSTITUTEDAMINO)-1,3,5-DITHI-AZINO]IMIN-11-(PIPRAZINE-1 YL) DIBENZO[B,F][1,4] OXAZEPINES

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ABSTRACT

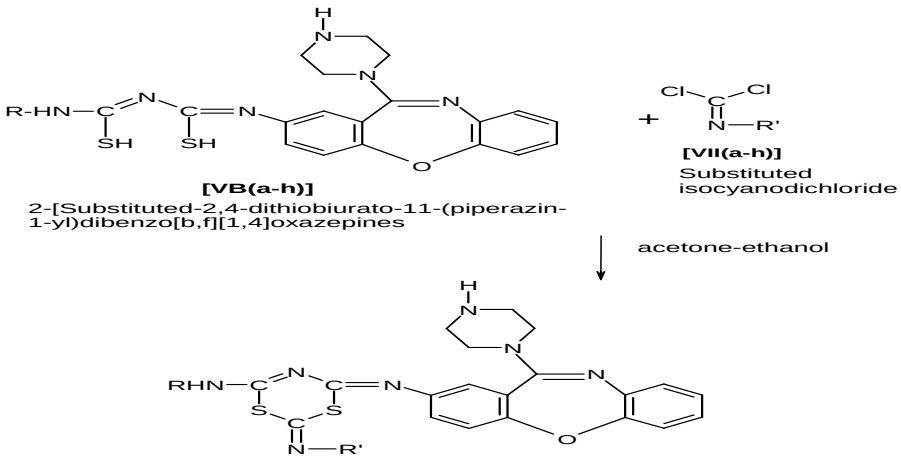
Recently in this laboratory convenient method for synthesis of 2-[(2-phenylimino-6-substitutedamino)-1,3,5-dithiazino]imino-11-(piprazine-1-yl) dibenzo [b,f] [1,4] oxazepines [**VIIIB(a-h)]** was developed. The interactions of 2-[substituted-2,4-dithiabiureto]-11-(piperazin-1-yl)dibenzo[b,f][1,4]oxazepines **[VB(a-h)]** with phenylisocyanodichloride **(VIIa)** in 50% acetone-ethanol medium were carried out on water bath to synthesized **[VB(a1-a36)]** respectively. The structures of synthesized compounds were justified on the basis of chemical characteristics, elemental analysis and spectral studies.

KEYWORDS: Phenylisothiocyanate, 2-[substituted-2,4-dithiabiureto]-11-(piperazin-1-yl) dibenzo[b,f][1,4]oxazepines and 50% acetone-ethanol.

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-h)]**. These were synthesized by the interactions 2-[substituted-2,4-dithiabiureto]-11-(piperazin-1-yl)dibenzo[b,f][1,4]oxazepines**[VB(a1-a36)]** and phenylisocyanodichloride **(VIIa)** in 50% acetone-ethanol medium **Scheme-1.**

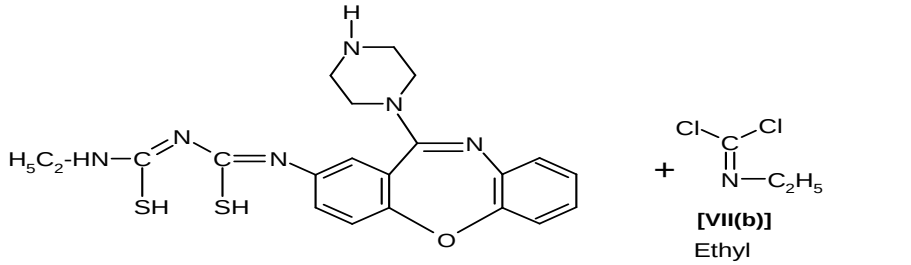


Where, R= -methyl, -ethyl, -tert-butyl, -phenyl, p-chlorophenyl, -p-tolyl.

Scheme-1

Synthesis of 2-[(2-ethylimino-6-ethylamino)-1,3,5-dithiozino]imino-11-(pipirazine -1-yl)dibenzo[b,f][1,4]oxazepine [VIIIB(a2)]

Synthesis of 2-[(2-ethylimino-6-ethylamino)-1,3,5-dithiozino]imino-11-(piprazine-1-yl) dibenzo[b,f][1,4]oxazepine was carried out by the interaction of 2,4-dithia- biureto-11-(piperazin-1-yl)dibenzo [b,f][1,4] oxazepine **[VB(a)]** with phenyl isocyano dichloride **(VIIa)** in 1:1 molar ratio. The interaction was carried out on in 50% acetone-ethanol medium for 4 hours on water bath. During refluxing evolution of hydrochloride gas was clearly noticed. After distillation of excess solvent, ivory colour product was isolated this on basification with dilute ammonium hydroxide brown crystals were afforded. Recrystalised from aqueous ethanol. Yield 90 %, m.p. 230˚C. The formation of **[VIIIB(a2)]** is depicted below.

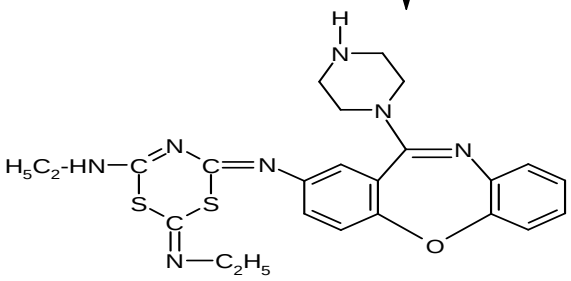


[VB(b)]
2-[Ethyl-2,4-dithiobiurato-11-(piperazin-1-yl)dibenzo[b,f][1,4]oxazepine

+

[VII(b)]
Ethyl isocyanodichloride

acetone-ethanol



[VIIIB(a2)]
2-[(2-Ethylimino-6-ethylamino)-1,3,5-dithiozino] imino-11-(piperazin-1-yl)dibenzo[b,f][1,4]oxazepine
[VIIIB(a2)]

Properties of [VIIIB(a2)]

It is brown colour crystalline solid having melting point 230^0C. 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: 65.70% (found), 66.11% (calculated)], [H: 03.50% (found), 04.58% (calculated)], [N: 16.63% (found), 16.63 % (calculated)], [S: 09.86% (found), 10.86% (calculated)]. **IR Spectrum**: The IR spectrum was carried out in KBr-pellets The important absorptions are correlated as (cm^{-1}) 3390.86 N–H stretching, 2891.30 C-H stretching, 2088.91 -S-C=N stretching, 1620.21 -C=N imino stretching, 1286.52 C-N stretching, 0794.67 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 9.7816-6.9430 ppm, -NH proton at δ 4.6843-3.3459 ppm, -CH_2 protons at δ 2.5270-2.1305 ppm, -CH_3 protons at δ 1.2352-1.1607 ppm.

Similarly,2-(phenyl-2,4-dithibiureto)-11-(piperazine-1-yl)dibenzo[b,f][1,4] oxazepine **[VA(a)],** 2-(methyl-2,4-dithibiureto)-11-(piperazine-1-yl)dibenzo[b,f] [1,4]oxazepine **[VA(c)],**2-(t-butyl-2,4-dithibiureto)-11-(piperazine-1-yl)dibenzo[b,f][1,4]oxazepine **[VA(d)],** 2-(p-chlorophenylthibiureto)-11-(piperazine-1-yl)dibenzo [b,f][1,4]oxazepine **[VA(e)],** 2-(p-tolyl-2,4-dithibiureto)-11-(piprazine-1-yl)dibenzo [b,f] [1,4]oxazepine **[VA(f)],** interacts with ethylisocyanodichloride **(VIIb)** by the above mentioned method respectively to isolate 2-[(2-ethylimino-6-phenylamino)-1,3,5-dithiozino] imino-11-(piperazine-1-yl)dibenzo[b,f][1,4]oxazepine **[VIIIB(b)]** 2-[(2-ethylimino-6-methylamino)-1,3,5-dithiozino]imino-11-(piperazine-1-yl) dibenzo [b,f] [1,4] oxazepine **[VIIIB(c)],** 2-[(2- ethylimino-6-tert-butylamino)-1,3,5-dithiozino]imino-11-(piperazine-1-yl)dibenzo[b,f] [1,4]oxazepine **[VIIIB(d)],** 2-[(2-ethylimino-6-p-chlorophenylamino)-1,3,5-dithiozino] imino-11-(piperazine-1-yl) dibenzo[b,f][1,4]oxazepine **[VIIIB(e)],** 2-[(2-ethylimino-6-p-tolylamino)-1,3,5-dithiozino]imino-11-(piperazine-1-yl)dibenzo[b,f] [1,4]oxazepine **[VIIIB(f)],** by the above mentioned method and enlisted in **Table No. I.**

Table No. I

Sr. No.	Compd. No	2-[(2-Ethylimino-6-substitutedamino)-1,3,5-dithiozino]imino-11-(piperazine-1-yl) dibenzo[b,f] [1,4] oxazepine	Yield (%)	m.pt. (^{0}C)
1.	[VIIIB(b)]	2-[(2-Ethylimino-6-ethylamino)----------oxazepine	92	240
2.	[VIIIB(c)]	2-[(2-Ethylimino-6-methylamino)----------oxazepine	88	286
3.	[VIIIB(d)]	2-[(2-Ethylimino-6-t-butylamino)---------- oxazepine	92	242
4.	[VIIIB(e)]	2-[(2-Ethylimino-6-p-chlorophenylamino)---oxazepine	85	290
5.	[VIIIB(f)]	2-[(2-Ethylimino-6-p-tolylamino)----------oxazepine	86	295

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