

**SYNTHESIS OF 2-[(2-METHYLDIMINO-6-SUBSTITUTEDAMINO)-1,3,5-DITHIAZINO]IMINO-11-(PIPAZINE-1-YL) DIBENZO[b,f][1,4] OXAZEPINES**

P. R. Kale<sup>1</sup>, D. T. Tayade<sup>2\*</sup>, A. S. Shendge<sup>2</sup>, K. S. Panpaliya<sup>2</sup>

<sup>1</sup>Department of Chemistry, S.R.R.Lahoti Science College, Morshi 444905.

<sup>2</sup>Department of Chemistry, Government Vidarbha Institute of Science and Humanities, Amravati 444604.

\*Corresponding Author: D. T. Tayade

Department of Chemistry, Government Vidarbha Institute of Science and Humanities, Amravati 444604.

Article Received on 21/04/2016

Article Revised on 12/05/2016

Article Accepted on 31/05/2016

**ABSTRACT**

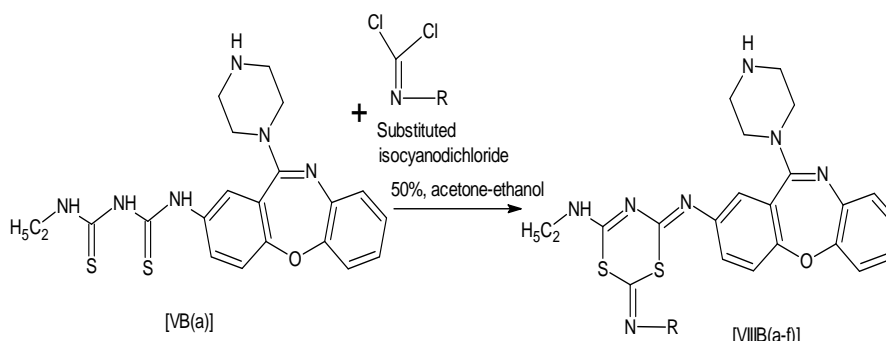
Recently synthesis of 2-[(2-methylimino-6-substitutedamino)-1,3,5-dithiazino] imino-11-(piperazine-1-yl) dibenzo [b,f] [1,4] oxazepines [VIIIb(a-f)] were carried out by the interactions of 2-[ethyl-2,4-dithiabiureto]-11-(piperazin-1-yl) dibenzo [b,f] [1,4] oxazepine [VB(a)] with substitutedisocyanodichlorides (VIIa-f) in 50% acetone-ethanol medium on water bath in good yield. The structure determination of synthesized compounds was done on the basis of elemental analysis, chemical characteristics and spectral studies.

**KEYWORDS:-** Substitutedisocyanodichlorides, 2-[ethyl-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<sup>[1]</sup> such as enzyme inhibitors<sup>[2]</sup>, analgesic<sup>[3]</sup>, anti-depressant<sup>[4]</sup> and psychoactive drugs.<sup>[5]</sup> Oxazepine nucleus is used for treatment of depression, anxiety and agitation.<sup>[6-7]</sup> 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<sup>[8-11]</sup> 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. We synthesized a novel series of 2-[(2-ethylimino-6-substitutedamino)-1,3,5-dithiazino]imino-11-(piperazine-1-yl)dibenzo [b,f] [1,4] oxazepines [VIIIb(a-f)]. These were synthesized by the interactions 2-[ethyl-2,4-dithiabiureto]-11-(piperazin-1-yl) dibenzo[b,f] [1,4]oxazepines [VB(a)] and substitutedisocyanodichlorides (VIIa-f) in 50% acetone-ethanol medium **Scheme-1**.



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

**Scheme-1**

**Synthesis of 2-[(2-methylimino-6-ethylamino)-1,3,5-dithiazino]imino-11-(piperazine-1-yl)dibenzo[b,f][1,4]oxazepine [VIIIb(a)]**

Synthesis of 2-[(2-ethylimino-6-ethylamino)-1,3,5-dithiazino]imino-11-(piperazine-1-yl)dibenzo[b,f][1,4]oxazepine was carried out by the interaction of 2-[methyl-2,4-dithiabiureto]-11-(piperazin-

1-yl)dibenzo[b,f][1,4]oxazepine[VB(a)] with phenylisocyanodichloride (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. Recrystallised from aqueous ethanol. Yield 90 %, m.p. 230°C.

#### Properties of [VIIIb(a)]

It is brown colour crystalline solid having melting point 230°C. 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<sub>6</sub> while insoluble in carbon tetrachloride, chloroform, benzene, petroleum ether. It formed picrate having melting point 209°C. **Elemental analysis:** [C: 58.20% (found), 58.41% (calculated)], [H: 05.30% (found), 05.47% (calculated)], [N: 19.64% (found), 19.87% (calculated)], [S: 12.86% (found), 12.98% (calculated)]. **IR Spectrum:** The IR spectrum was carried out in KBr-pellets. The important absorptions are correlated as (cm<sup>-1</sup>) 31836.73 N-H stretching, 2738.25 C-H stretching, 2152.64 -S-C=N stretching, 1632.31 -C=N imino stretching, 1247.49 C-N stretching, 8010.94 C-S stretching. **NMR Spectrum:** The NMR spectrum was carried out in DMSO-d<sub>6</sub> and CDCl<sub>3</sub>, this spectrum distinctly displayed the signals due to Ar-H protons at  $\delta$  7.7814-6.3638 ppm, -NH proton at

$\delta$  2.6546-2.1375 ppm, -CH<sub>2</sub> protons at  $\delta$  2.4127-2.1202 ppm, -CH<sub>3</sub> protons at  $\delta$  1.1607 ppm.

Similarly, 2-(methyl-2,4-dithiureto)-11-(piperazine-1-yl)dibenzo[b,f][1,4]oxazepine [VA(a)] was interacted with p-chlorophenylisocyanodichloride (VIb), ethylisocyanodichloride (VIc), methylisocyanodichloride (VI d), t-butylisocyanodichloride (VIe), p-tolyl isocyanodichloride (VI f) by the above mentioned method respectively to isolate 2-[(2-methylimino-6-phenylamino)-1,3,5-dithiozino]imino-11-(piperazine-1-yl)dibenzo[b,f][1,4] oxazepine [VIIIb(b)] 2-[(2-methylimino-6-methylamino)-1,3,5-dithiozino]imino-11-(piperazine-1-yl) dibenzo [b,f] [1,4] oxazepine [VIIIb(c)], 2-[(2-methylimino-6-tert-butylamino) -1,3,5-dithiozino]imino-11-(piperazine-1-yl)dibenzo[b,f][1,4]oxazepine [VIIIb(d)], 2-[(2-methylimino-6-p-chlorophenylamino)-1,3,5-dithiozino]imino-11-(piperazine-1-yl)dibenzo [b,f][1,4]oxazepine [VIIIb(e)], 2-[(2-methylimino-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-Methylimino-6-substitutedamino)-1,3,5-dithiozino]imino-11-(piperazine-1-yl) dibenzo[b,f] [1,4] oxazepine	Yield (%)	m.pt. (°C)
1.	[VIIIb(b)]	2-[(2-Methylimino-6-ethylamino)-----oxazepine	92	240
2.	[VIIIb(c)]	2-[(2-Methylimino-6-methylamino)-----oxazepine	88	286
3.	[VIIIb(d)]	2-[(2-Methylimino-6-t-butylamino)----- oxazepine	92	242
4.	[VIIIb(e)]	2-[(2-Methylimino-6-p-chlorophenylamino)---oxazepine	85	290
5.	[VIIIb(f)]	2-[(2-Methylimino-6-p-tolylamino)-----oxazepine	86	295

#### REFERENCES

1. Tripathi D.N., Malhotra RC. Bhattacharya A., J. Chromatography, 1984; 315: 417.
2. Levinspial O., Chem. Rea. Engg, John Willey and Sons 2<sup>nd</sup> Ed, 1995.
3. Aiello, F.; Brizzi, A.; Garofalo, A.; Grande, F.; Ragno, G.; Dayam, R.; Neamati, N. Bioorg. Med. Chem, 2004; 12: 4459.
4. Pecher J., Waefelaer A. Bull. Soc. Chim. Belg, 1978; 87: 911.
5. Halina K., Malgorzata S., Agata, W., 2012; 9(6); 828-850.
6. Ayab H. Journal of Al-Nahrain, 2012; 15(4): 47-59.
7. J. Mikim, K. Y. Lee and J. N. kim. Bull. Korean Chem., 2002; 23(8): 1055-1056.
8. Bansal R.K., J. Heterocyclic Chemistry, 2012; 8: 12-24.
9. Fernandes P.S and Sonar T.M., J.Ind. Chem. Soc., 1986; 53(4): 427.
10. Saleem F., Eur. Pat., CHAPPL 87/1 APR, 1987; 13: 3600009. Chem Abstr, 1989; 110: 114893.
11. Hedge J.C., Satheesha Rai N. and Balkrishna K., J.Chem. Sci., III 2007; 9(4): 299-302.