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SYNTHESIS OF 2-[SUBSTITUTED-2,4-DITHIABIURETO]-11-(PIPERAZIN-1-YL) DIBENZO [b,f][1,4] OXAZEPINES

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ABSTRACT

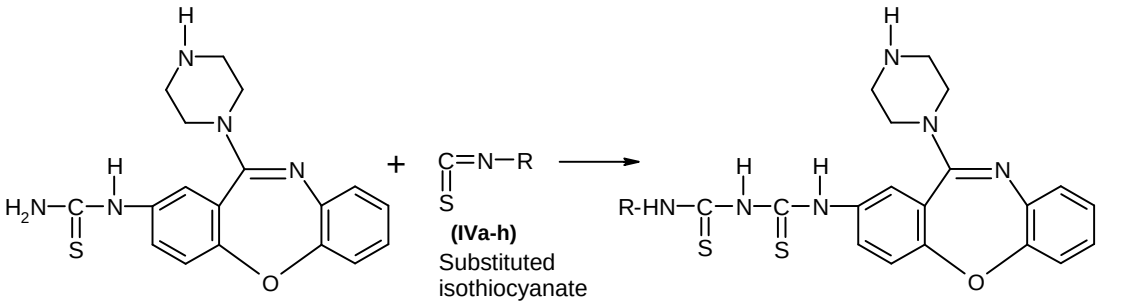
*Recently in this laboratory convenient method for synthesis of 2-[substituted-2,4-dithiabiureto]-11-(piperazin-1-yl)dibenzo[b,f][1,4]oxazepines **[VB(a-h)]** was developed. The interactions of 2-thiocarbamido-11-(piperazin-1-yl)dibenzo[b,f][1,4]oxazepine **[IIIB(a)]** with various isothiocyanates **(IVa-h)** in 50% acetone-ethanol medium were carried out on water bath to synthesized **[VB(a-h)]** respectively. The structures of synthesized compounds were justified on the basis of chemical characteristics, elemental analysis and spectral studies.*

Keywords:- Various isothiocyanate, 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-[substituted-2,4-dithia- biureto]-11-(piperazin-1-yl)dibenzo[b,f][1,4]oxazepines **[VB(a-h)].** These were synthesized by the interactions 2-thiocarbamido-11-(piperazin-1-yl) dibenzo [b,f][1,4]-oxazepine **[IIIB(a)]** with various isothiocyanates **(IVa-h)** in 50% acetone-ethanol medium **Scheme-1.**



[IIIB(a)]
2-[Thiocarbamido-11-(piperazin-1-yl)dibenzo [b,f][1,4]oxazepine

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

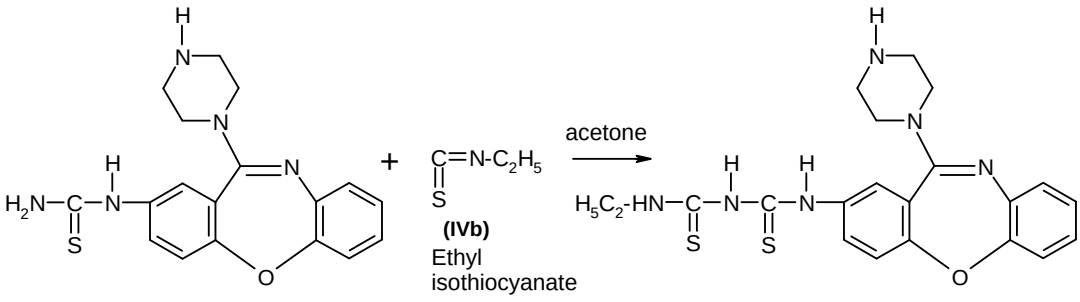
Where, R= -methyl, -ethyl, -t-butyl, -phenyl, p-chlorophenyl, -p-tolyl.

Scheme-1

Synthesis of 2-[Ethyl-2,4-dithiabiureto]11-(piperazin-1-yl) dibenzo [b,f] [1,4] oxazepine

A reaction mixture of 2-thiocarbamido-11-(piperazin-1-yl) dibenzo [b,f][1,4] oxazepine **[IIIB(a)]** and ethylisothiocyanate **(IVb)** in 1:1 molar proportion were refluxed in 50% acetone-ethanol medium for 4 hours on water bath, brown colour crystals were separated out, they were filtered and dried at room conditions.

Recrystalised from aqueous ethanol**.** Yield 84 %, m.p. 168^0C. The formation of **[VA(a)]** is depicted below,



[IIIB(a)]
2-[Thiocarbamido-11-(piperazin-1-yl)dibenzo [b,f][1,4]oxazepine

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

[VA(a)]

Properties of [VB(b)]

It is brown colour crystalline solid having melting point 168^0C. 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 120^0C.

Elemental Analysis: The result of elemental analysis is given in **Table No. 1**

Table No. 1

Sr.No.	Elements	Found	Calculated
1.	Carbon	56.18	57.27
2.	Hydrogen	04.84	05.85
3.	Nitrogen	18.85	19.09
4.	Sulphur	13.36	14.54

IR spectrum: IR spectrum of compound **[VB(a)]** was carried out in KBr-pellets and is reproduced on **IR Plate No. PRK-** The specific absorption is correlated as follows and is depicted in **Table No.2**

Table No. 2

Sr.No.	Absorption Observed (cm^1)	Assignment	Absorption Expected (cm^1)
1.	3381.21	N-H stretching	3500-3000
2.	2899.01	C-H stretching	3000-2500
3.	1614.42	N=C-N stretching	1750-1180
4.	1519.91	N-C=S stretching	1550-1250
5.	1224.80	C-O-C streching	1300-1100
6.	1112.93	C-C streching	1120-1100
7.	1012.63	C-N stretching	1200-1000

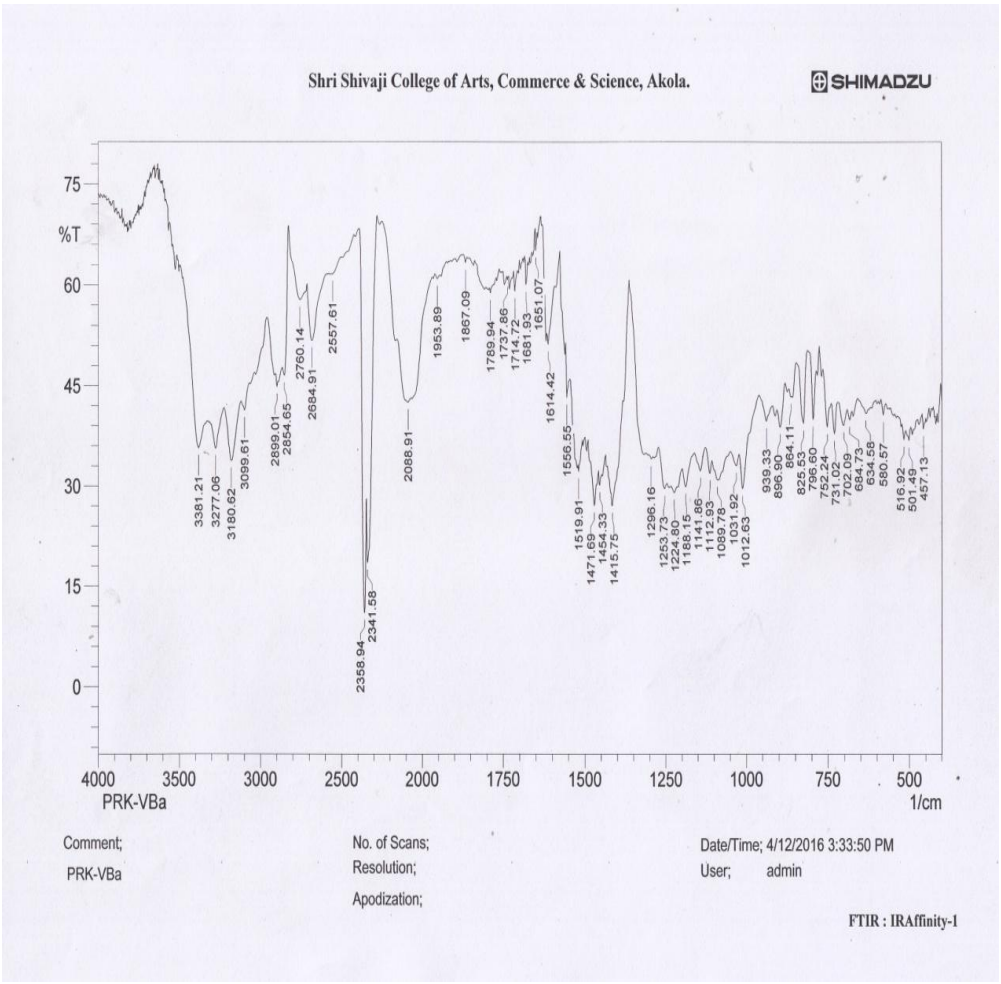
NMR Spectrum: The NMR spectrum was carried out in DMSO-d_6 and $CDCl_3$ This spectrum distinctly displayed the signals due to Ar-H protons at δ 9.3773-6.9786 ppm, -NH protons at δ 5.1161-3.3360 ppm, -CH_2 protons at δ 2.5400-2.5269 ppm, -CH_3 protons at δ 1.2430 ppm.

Similarly, 2-thiocarbamido-11-(piperazin-1-yl)dibenzo[b,f][1,4]oxazepine **[IIIB(a)]** interact with phenylisothiocyanate **(IVb)** methylisothiocyanate **(IVc)** t-butylisothiocyanate **(IVd)** p-chorophenylisothiocyanate **(IVe)** p-tolylisothiocyanate **(IVf)** to form 2-[methyl-2,4-dithiabiureto]11-(piperazin-1-yl)dibenzo-[b,f][1,4]oxazepine**[VB(c)],**2-[tert-butyl-2,4-dithia- biureto]11-(piperazin-1-yl) dibenzo-[b,f] [1,4] oxazepine **[VB(d)],** 2-[p-chlorophenyl-2,4-dithiabiureto]11-(piperazin-1-yl)dibenzo-[b,f][1,4oxazepine **[VB(e)],** 2-[o-tolyl-2,4-dithiabi-ureto]11-(piperazin-1-yl)dibenzo-[b,f][1,4]oxazepine **[VB(f)],** 2-[m-tolyl-2,4-dithiabiureto] 11-(piperazin-1-yl)dibenzo-[b,f][1,4] oxazepine **[VB(g)],** 2-[p-tolyl-2,4-dithiabiureto]11-(piperazin-1-yl)dibenzo-[b,f] [1,4] oxazepine **[VB(h)]** respectively by the above mentioned method and enlisted in **Table No. 3**

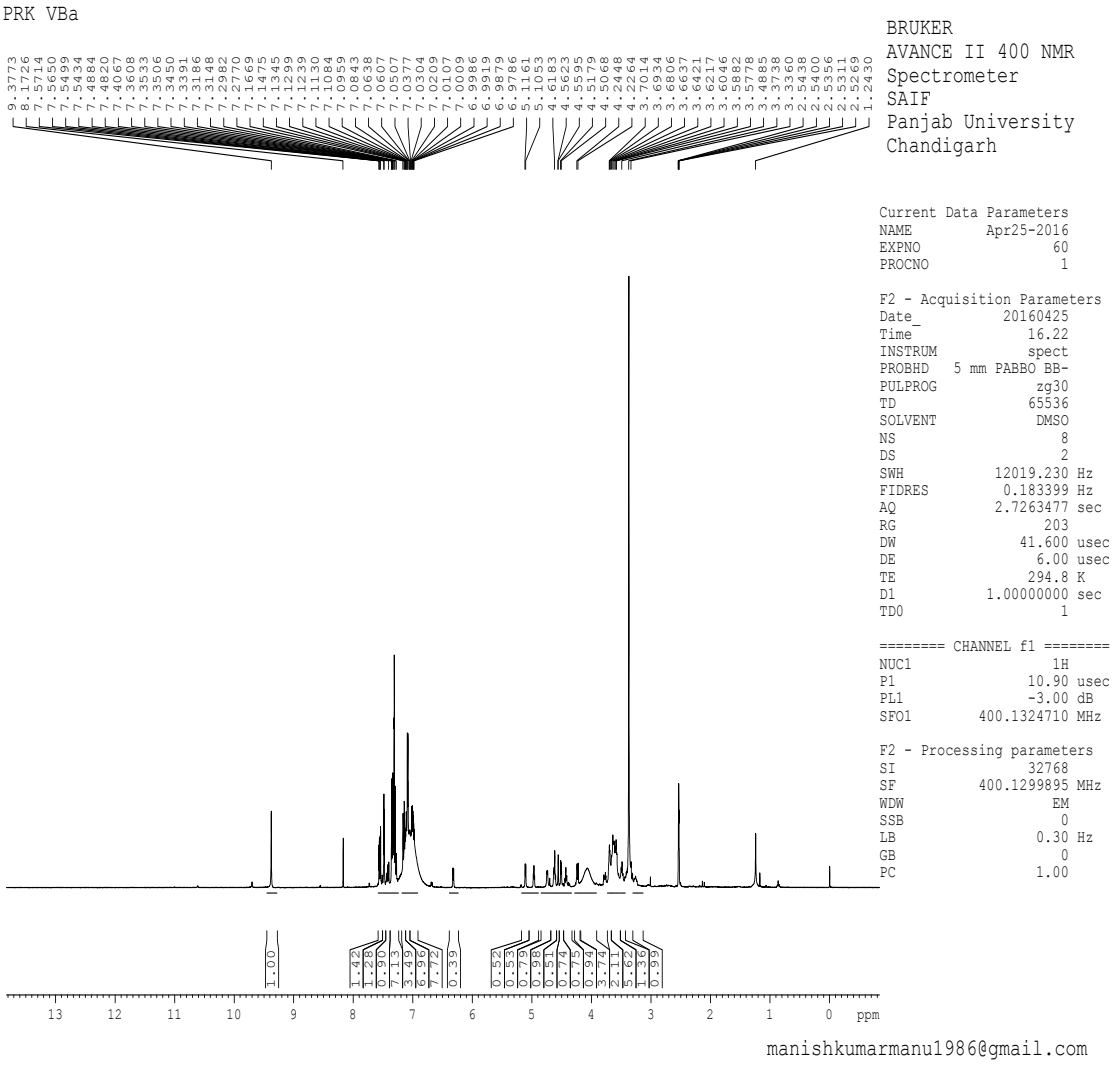
Table No. 3

Sr. No.	2-[**Substituted**-2,4-dithiabiureto]11(piperazin-1-yl dibenzo-[b,f][1,4]oxazepine **[VB(c-h)]**	**Yield (%)**	**M.P. ˚C**
1.	2-[**phenyl**----------oxazepine **[VB(c)]**	86	219
2.	2-[**Methyl**----------oxazepine **[VB(c)]**	90	176
3.	2-[**Tert-butyl**----------oxazepine **[VB(d)]**	94	133
4.	2-**p-Chlorophenyl**------oxazepine **[VB(d)]**	86	243
5.	2-[**p-Tolyl**----------oxazepine **[VB(e)]**	91	237

IR Spectra of VBa



NMR Spectra of VBa



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