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SYNTHESIS OF 8-[(2-ETHYLIMINO-6-SUBSTITUTEDAMINO)-1,3,5-DITHIAZINO- IMINO]-1-METHYL-6-PHENYL-4H-[1,2,4]AZOLO [4,3-a] [1,4] BENZODIAZEPINES

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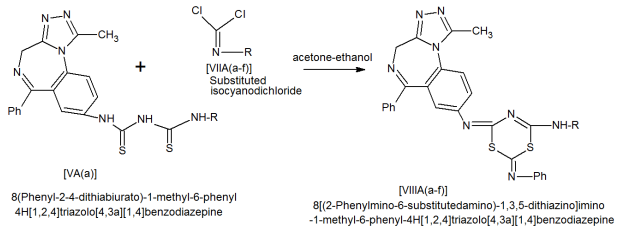
ABSTRACT

Recently in this laboratory a novel series of 8-[(2-ethylimino-6-substitutedamino)-1,3,5-dithiazino]imino-1-methyl-6-phenyl-4H-[1,2,4]triazolo[4,3a][1,4]benzodiazepine was synthesized by the interactions of 8-(ethyl-2,4-dithibiureto)-1-methyl-6-phenyl-4H-[1,2,4] triazolo [4,3-a][1,4]benzodiazepine **[VA(a)]** with substituted isocyanodichlorides **(VIIa-f)** in 50% acetone-ethanol medium. The structure determination of synthesized compounds was done on the basis of elemental analysis, chemical characteristics and spectral studies.

INTRODUCTION

1,4-Benzodiazepinedione has been reported as potent antagonist's interaction in vitro and in cell-based assays and also proved that they possess anti-conversant, anxiolytic, anti-tumor properties[1]. It is effective against cholecystokinin receptor (CCK), opiate receptor and platelet glycoprotein antagonists[2-3]. Many derivatives of benzodiazepines are widely used as sedative, anti-depressive, anti-inflammatory and hypnotic agents[4-5]. It is also used as dyes for acrylic fibers[6]. 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[7-10] were studied. [1,4] Benozodiazepine, 2,4-dithiobiurets, 1,3,5-dithiazines and their derivatives showed agricultural, medicinal, biological, pharmaceutical, industrial significances and applications. By considering all these facts this research scheme was designed to synthesized a novel series of 8-[(2-ethylimino-6-substituted amino)-1,3,5-dithiazino]imino-1-methyl-6-phenyl-4H-[1,2,4] triazolo [4,3a][1,4] benzodiazepines **Scheme-I.**



8(Phenyl-2-4-dithiabiurato)-1-methyl-6-phenyl 4H[1,2,4]triazolo[4,3a][1,4]benzodiazepine

8[(2-Phenylmino-6-substitutedamino)-1,3,5-dithiazino]imino -1-methyl-6-phenyl-4H[1,2,4]triazolo[4,3a][1,4]benzodiazepine

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

Scheme-I Synthesis of 8-[(2-ethylimino-6-phenylamino)-1,3,5-dithiazinoimino]-1-methyl-6-phenyl-4H -[1,2,4]triazolo[4,3-a][1,4]benzodiazepine[VIIIA(a)]

Interactions of 8-(ethylimino-2,4-dithibiureto)-1-methyl-6-phenyl-4H-[1,2,4] triazolo [4,3-a] [1,4]benzodiazepine **[VA(a)]** with phenylisocyanodichloride **(VIIa)** in 1:1 molar ratio was carried out 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 then brown crystals were afforded. Recrystalised from aqueous ethanol. Yield 91%, m.p. 237˚C.

Properties of [VIIIA(a)]

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Table-1

Sr. No	Compd. No	8-[(2-Ethylimino-6-**substituted**amino)-1,3,5-dithiozino]imino-1-methyl-6-phenyl-4H-[1,2,4] triazolo [4,3-a] [1,4] benzodiazepine	Yield (%)	m.pt. (^{0}C)
1	**[VIIIA(b)]**	8-[(2-Ethylimino-6-**ethyl**amino)----------benzodiazepine	78	125
2	**[VIIIA(c)]**	8-[(2-Ethylimino-6-**methyl**amino)----------benzodiazepine	81	109
3	**[VIIIA(d)]**	8-[(2-Ethylimino-6-t-**butyl**amino)----------benzodiazepine	93	147
4	**[VIIIA(e)]**	8-[(2-Ethylimino-6-**p-chlorophenyl**amino)------benzodiazepine	84	214
5	**[VIIIA(f)]**	8-[(2-Ethylimino-6-**p-tolyl**amino)----------benzodiazepine	69	209

It is lemon colour crystalline solid having melting point 237^{0}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_6 while insoluble in carbon tetrachloride, chloroform, benzene, petroleum ether. It formed picrate having melting point 207^{0}C. **Elemental analysis: [**C: 61.93% (found), 62.68% (calculated)], [H: 04.37% (found), 04.47% (calculated)], [N: 20.17% (found), 20.89% (calculated], [S: 11.50% (found), 11.94% (calculated)]. **IR Spectrum**: The IR spectrum was carried out in KBr-pellets. The important absorptions are correlated as (cm^{-1}): 3267.43 N–H stretching, 2794.62 C-H stretching, 2054.72 -S-C=N stretching, 1635.93 N=C-N stretching, 1123.87 C-N stretching, 635.32 C-S stretching. **NMR Spectrum:** The NMR spectrum was carried out in DMSO-d_6 and $CDCl_3$, Ar-H protons at δ 7.1548-6.1734 ppm, -NH proton at δ 3.2185-3.0623 ppm, -CH_2 protons at δ 2.1947-2.0264 ppm, -CH_3 protons at δ 1.1634 ppm.

Similarly, 8-(ethylimino-2,4-dithibiureto)-1-methyl-6-phenyl-4H-[1,2,4] triazolo [4,3-a] [1,4]benzodiazepine **[VA(a)]** interact with p-chlorophenylisocyanodichloride **(VIb),** ethyl isocyanodichloride **(VIc),** methylisocyanodichloride **(VId),** t-butylisocyanodichloride **(VIe),** p-tolylisocyanodichloride **(VIf),** by the above mentioned method respectively to isolate 8-[(2-ethylimino-6-p-chlorophenylamino)-1,3,5-dithiazino]imino-1-methyl-6-phenyl-4H[1,2,4] triazolo[4,3-a][1,4] benzodiazepine **[VIIIA(b)],** 8-[(2-ethylimino-6-ethylamino)-1,3,5-dithi azino]imino-1-methyl-6-phenyl-4H-[1,2,4]triazolo[4,3-a][1,4]benzodiazepine **[VIIIA(c)],** 8-[(2-ethylimino-6-methylamino)-1,3,5-dithiazino]imino-1-methyl-6-phenyl-4H-[1,2,4]-tri- azolo [4,3-a] [1,4] benzodiazepine **[VIIIA(d)],** 8-[(2-ethylimino-6-t-butylamino)-1,3,5-dithi- azino]imino-1-methyl-6-phenyl-4H-[1,2,4]triazolo[4,3-a][1,4]benzodiazepine**[VIIIA(e)],** 8-[(2-ethylimino-6-p-tolylamino)-1,3,5-dithiazino]imino-1-methyl-6-phenyl-4H[1,2,4]triazolo- [4,3a][1,4]benzodiazepine **[VIIIA(f)]** by the above mentioned method and enlisted in **Table-1**

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