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RESEARCH ARTICLE

SYNTHESIS OF 8-[(2-p-CHLOROPHENYLIMINO-6-SUBSTITUTEDAMINO)-1,3,5-DITHIAZINOIMINO]-1-METHYL-6-PHENYL-4H[1,2,4]AZOLO[4,3-a] [1,4] BENZODIAZEPINES

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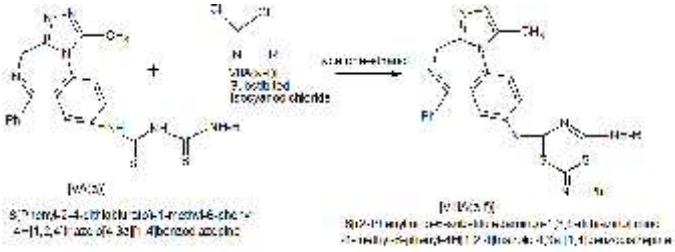
Various isocyanodichlorides, 8- (p-chlorophenyl-2,4-dithiabiureto)-1-methyl-6-phenyl-4H-[1,2,4] triazolo [4,3-a] [1,4] benzodiazepines and 50% acetone-ethanol.

ABSTRACT

A novel series of 8-[(2-p-chlorophenylimino-6-substitutedamino)-1,3,5-dithiazino]imino-1-methyl-6-phenyl-4H-[1,2,4]triazolo[4,3a][1,4]benzodiazepines **[VIIIA(a-f)]** had been synthesized by refluxing 8-(p-chlorophenyl-2,4-dithibiureto)-1-methyl-6-phenyl-4H-[1,2,4] triazolo- [4,3-a][1,4]benzodiazepines **[VA(a)]** with various isocyanodichlorides **(VIIa-f)** in 50% acetone-ethanol medium in 1:1 molar proportion. The structures of all synthesized compounds were justified on the basis of chemical characteristics, elemental analysis and spectral studies.

INTRODUCTION

1,4-Benzodiazepine nucleus containing molecule showed anti-conversant, anxiolytic, anti-tumor properties[1]. This is also effective against cholecystokinin receptor (CCK), opiate receptor and platelet glycoprotein antagonists[2-3]. Various 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 significances and applications in life as well as agricultural sciences so various 8-[(2-p-chlorophenylimino-6-substitutedamino)-1,3,5-dithiazino]imino-1-methyl-6-phenyl-4H-[1,2,4] triazolo[4,3a] [1,4]benzodiazepines **[VIIIA(a-f)]** were synthesized by the interactions of 8-(p-chlorophenyl-2,4-dithiabiureto)-1-methyl-6-phenyl-4H-[1,2,4]triazolo[4,3-a] [1,4] benzodiazepines **[VA(a-h)]** with various isocyanodichlorides **(VIIa-f)** in 50% acetone-ethanol medium, **Scheme-I.**



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

Scheme-I

Synthesis of 8-[(2-p-chlorophenylimino-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-(p-chlorophenylimino-2,4-dithibiureto)-1-methyl-6-phenyl-4H-[1,2,4] triazolo [4,3-a] [1,4]benzodiazepine **[VA(a)]** with phenylisocyanodichloride **(VIIa)** 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 93%, m.p. 237^0C.

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Table No.1

Sr. No	Compd. No	8-[(2-p-Chlorophenylimino-6-substitutedamino) -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-p-Chlorophenylimino-6-**ethyl**amino)----------benzodiazepine	74	201
2	**[VIIIA(c)]**	8-[(2-p-Chlorophenylimino-6-**methyl**amino)----------benzodiazepine	89	217
3	**[VIIIA(d)]**	8-[(2-p-Chlorophenylimino-6-t**ert-butyl**amino)----------benzodiazepine	67	184
4	**[VIIIA(e)]**	8-[(2-p-Chlorophenylimino-6-**p-chlorophenyl-** amino)----------benzodiazepine	91	246
5	**[VIIIA(f)]**	8-[(2-p-Chlorophenylimino-6-**p-tolyl**amino)----------benzodiazepine	84	219

Properties of [VIIIA(a)]

It is brown colour crystalline solid having melting point 237^{0}C. It gave positive test for nitrogen, sulphur and chlorine. 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 217^{0}C. **Elemental analysis: [**C: 61.84% (found), 62.08% (calculated), H: 03.62% (found), 03.71% (calculated), N: 18.10% (found), 18.10% (calculated, S: 09.50% (found), 10.34% (calculated) Cl: 5.36% (found), 5.73% (calculated)]. **IR Spectrum**: The IR spectrum was carried out in KBr-pellets. The important absorptions are correlated as (cm^{-1}): 3474.53 N–H stretching, 2794.57 C-H stretching, 2032.45 -S-C=N stretching, 1593.69 N=C-N stretching, 1135.72C-N stretching. 7194.26 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 7.7573-6.264 ppm, -NH proton at δ 3.2592-3.1427 ppm, -CH_2 protons at δ 2.53521-2.3214 ppm, -CH_3 protons at δ 1.1734 ppm.

Similarly,8-(p-chlorophenyl-2,4-dithiabiureto)-1-methyl-6-phenyl-4H-[1,2,4]tri- azolo [4,3-a][1,4] benzodiazepine **[VA(b)]** was interacted with p-chlorophenylisocyano-dichloride **(VIb),** ethylisocyanodichloride **(VIc),** methylisocyanodichloride **(VId),** t-butyliso- cyanodichloride **(VIe),** p-tolylisocyanodichloride **(VIf)** by the above mentioned method respectively to isolate 8-[(2- p-chlorophenylimino-6-ethylamino)-1,3,5-dithiazino]imino-1-methyl-6-phenyl-4H-[1,2,4] triazolo[4,3-a][1,4] benzodiazepine **[VIIIA(b)],** 8-[(2-p-chlorophenylimino-6-methylamino)-1,3,5-dithiazino]imino-1-methyl-6-phenyl-4H-[1,2,4]tri azolo[4,3-a][1,4] benzodiazepine **[VIIIA(c)],** 8-[(2-p-chlorophenylimino-6-t-butylamino)-1,3,5-dithiazino]imino-1-methyl-6-phenyl-4H-[1,2,4] triazolo[4,3-a] [1,4] benzodiazepine **[VIIIA(d)],** 8-[(2-p-chlorophenylimino-6-p-chlorophenylamino)-1,3,5-dithiazino]imino-1-methyl-6-phenyl-4H-[1,2,4]triazolo[4,3-a][1,4]benzodiazepine **[VIIIA(e)],** 8-[(2- p-chlorophenylimino-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 No.1**

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