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Synthesis of N-[7-(1-Substituted) -2,4-Dithiobiureto)-4-YL]-N,N-Diethyl-Pentane-1,4-Diamine

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ABSTRACT

Heteroacyclic and Heterocyclic containing drugs showed remarkable and noticeable drug absorption, transmission and drug effects; hence they created their own identity and importance in pharmaceutical, medicinal, agricultural and drug sciences. Thioamido, pyridino, thiobiureto and alkylamino heterocyclic compounds showed various significances and applications in industrial, pharmaceutical, medicinal and drug chemistry. Considering all these facts into consideration it was thought interesting to synthesize N-[7-(1-substituted)-2,4-dithiobiureto)-4-yl]-N,N-diethyl-pentane-1,4-diamine (5) by interacting N-(7-thiocarbamidoquinoline-4-yl)-N,N-diethyl-pentane-1,4-diamines (3) with various isothiocynates (4) in ethanol-acetone medium. The N-(7-thiocarbamidoquinoline-4-yl)-N,N-diethyl-pentane-1,4-diamines (3) was synthesized by interacting N-(7-chloroquinoline-4-yl)-N,N-diethyl-pentane-1,4-diamine (1) with thiourea (2) in isopropanol medium. The justification and identification of the structure of these newly synthesized compounds had been established on the basis of chemical characterization, elemental analysis, and through spectral data.

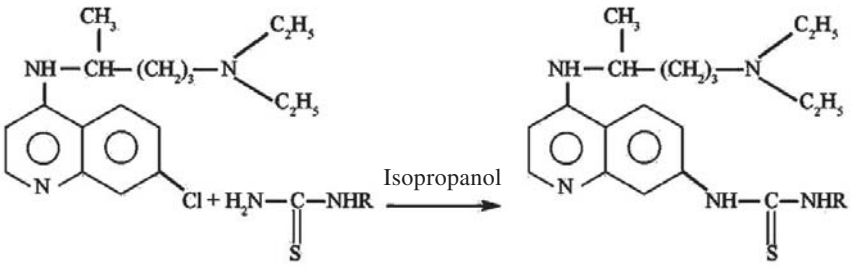
Key words: Substitutedisothiocynates, N-(7-substitutedthiocarbamidoquinoline-4-yl)-N, N-diethyl-pentane-1,4-diamines, Acetone and Ethanol.

INTRODUCTION

Recently in this laboratory, the synthetic applications of cyanoguanidine had been briefly explored.[1] As evident from the structure of the **N**-(7-thiocarbamidoquinoline-4-yl)-N,N-diethyl-pentane-1,4-diamines (3) , it was observed that there are various reactive sites in this molecule for the reactions. This molecule possesses –SH, and -NH_2 important reactive sites for the reactions. As a wider programmee of this laboratory in the synthesis of nitrogen, nitrogen and sulphur containing heteroacycles and heterocycles. The interactions of cyanoguanidine with various thiourea and alkyl or arylisothiocyanates have been investigated in sufficient details[2-5]. Some of these compounds showed remarkable pharmaceutical and biological activities[6]. The synthesized heteroacycles are used

as a best intermediated in the synthesis of thiadiazoles, dithiazoles, thiadizines, triazines, Hector's bases etc.

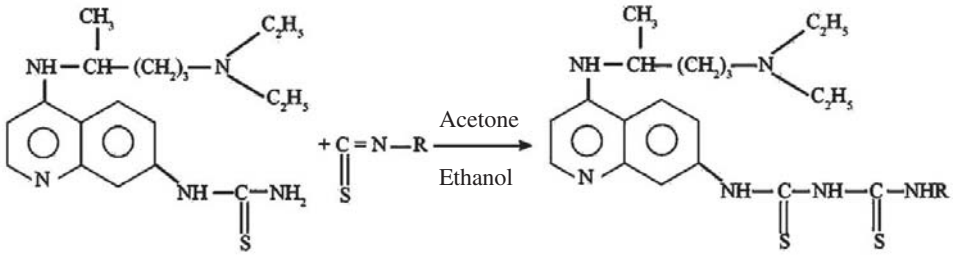
An exhaustive literature survey on substitutedbiureto and pyridino nucleus containing drugs created their own identity in medicinal and pharmaceutical sciences[7-8]. Hence taking all these things into considerations interaction of N-(7-chloroquinoline-4-yl)-N,N-diethyl-pentane-1,4-diamine (1) with thiourea (2) in isopropanol medium was investigated to synthesize N-(7-thiocarbamidoquinoline-4-yl)-N,N-diethyl-pentane-1,4-diamine(3). (Scheme-1) N-(7-thiocarbamidoquinoline-4-yl)-N,N-diethyl-pentane-1,4-diamines was then further interacted with alkyl or aryl isothiocynates (4) in acetone-ethanol medium to isolate yet new series of N-[7-(1-substituted) -2,4-dithiobiureto)-4-yl]-N,N-diethyl-pentane-1,4-diamine.(5) (Scheme-2)



(1) (2) (3)

Where R= -H, -phenyl, -methyl, -ethyl, -allyl

Scheme 1:



(3) (4) (5)

Where R= -H, -phenyl, -p-Cl-phenyl, -ethyl, t-butyl

Scheme 2:

EXPERIMENTAL

The melting point of all the synthesized compounds was recorded using hot Paraffin bath. The carbon and hydrogen analysis was carried out on Carlo-ebra 1106 analyzer. Nitrogen estimation was carried out on Colman-N-analyzer-29. IR spectra were recorded on Perkin Elmer Spectrometer in range 4000-400 cm^{-1} in KBr pellets PMR spectra were recorded on Brucker Ac 300 F spectrometer with TMS as internal slandered using $CDCl_3$ and DMSO–d_6 as solvent. The purity of compound was checked on silica Gel-G pellets by TLC with layer thickness of 0.3 mm. All chemicals used were of AR-grade.

N-(7-Thiocarbamidoquinoline-4-yl)-N, N-diethyl-pentane-1, 4-diamine (3a)

A mixture of N-(7-chloroquinoline-4-yl)-N, N-diethyl-pentane-1, 4-diamine (1) (0.1M), thiourea (2a) and isopropanol (40ml) was refluxed on boiling water bath for 4 hrs. During boiling suspended N-(7-chloroquinoline-4-yl)-N, N-diethyl-pentane-1, 4-diamine went into the solution and the new product

was found to be gradually separated out, which on basification with dilute ammonium hydroxide afforded white crystals. It was filtered in hot conditions and recrystallized with aqueous ethanol to obtained (3a), yield 67.7%, melting point 196^0 C. (D)

Properties

It is white, crystalline solid having melting point 196 ^{0}C. (D). It gave positive test for nitrogen and sulphur. Desulphurised with alkaline plumbite solution. It formed picrate, melting point 120 ^{0} C.

Elemental analysis

C [(found 67.4%) calculated 68.96], H [(found 7.17%) calculated 7.58%], N [(found 16.1%) calculated 16.19], S[(found 6.72%) calculated 7.35].

IR Spectra

The IR spectra was carried out in KBr pellets and The important absorption can be correlated as (cm^{-1}) 3435.6 (N-H stretching), [C-H(Ar)] stretching 3150.5 1638.1 (C-N stretching), 1523.6 (=C=NH imino), 1199.7(C-N stretching), 991.9 (N=C=S).

PMR Spectra

The spectrum was carried out in $CDCl_3$ and DMSO-d_6 .This spectrum distinctly displayed the signals due to Ar-H, protons at δ7.9525-7.9543 ppm. Ar-NH protons at δ 5.0417-5.0483 ppm, pyridino-NH at δ 4.2911-4.4564 ppm. –CH_2 protons at 3.3386-3.8638 ppm. –CH_3 protons at 1.2538ppm.

N-[7-(1- Phenyl) -2, 4-dithiobiureto)-4-yl]-N, N-diethyl-pentane-1, 4-diamine (5a)

A mixture of N-(7-thiocarbamidoquinoline-4-yl)-N, N-diethyl-pentane-1,4-diamines (3) (0.05M) and phenylisothiocyanate (4a) (0.05m) was refluxed on water bath in acetone-ethanol (20ml) medium for 4 hrs in round bottom flask. It was filtered in hot conditions. The resultant filtrate on distillation gave (5a), yield 72% m.p.146^{0}C.

Examination of Product

It is white crystalline solid having melting point 115^{0}C. It gave positive test for nitrogen and sulphur. Desulphurised with alkaline plumbite solution.

Elemental analysis

C [(Found 62.85%) calculated 63.15%], H [(found 5.95%) calculated 6.88%], N[(found 16.14%) calculated 17.00%], S[(found 11.28%) calculated 12.95%]

IR Spectrum

The IR spectrum was carried out in KBr pellets. The important absorption can be correlated as (cm^{-1}):- 3268.6 (N-H-Stretching), 3057.3 (Ar C-H-stretching), 2239.3(C=N stretching), 2935.0 (C-N-stretching), 1073.0 (C-S- stretching).

PMR Spectrum

The spectrum was carried out in $CDCl_3$ and DMSO-d_6 .This spectrum distinctly displayed the signals due to Ar-H, protons at δ7.1-7.5 ppm. Ar-NH protons at δ 6.9 ppm, pyridino-NH at δ 4.29-4.45 ppm. –CH_2 protons at 3.7 ppm. –CH_3 protons at 2.3-1.4ppm.

Similarly, N-[7-(1-p-Cl-phenyl) -2,4-dithiobiureto)-4-yl]-N,N-diethyl-pentane-1,4-diamine (5b), N-[7-(1-methyl) -2,4-dithiobiureto)-4-yl]-N,N-diethyl-pentane-1,4-diamine (5c), N-[7-(1-ethyl) -2,4-dithiobiureto)-4-yl]-N,N-diethyl-pentane-1,4-diamine (5d), N-[7-(1-allyl) -2,4-dithiobiureto)-4-yl]-N,N-diethyl-pentane-1,4-diamine (5e) were

Table 1:

S. No.	N-[7-(1- substituted) -2,4-dithiobiureto)-4-yl]-N,N-diethyl-pentane-1,4-diamine	Yield %	m.p. ^{0}C
1	…….-p-Cl-phenyl……	67	149
2	……..-methyl……	65	184
3	……-ethyl……..…..	71	181
4	……tert-butyl……..	59	161

synthesized by interacting N-(7-thiocarbamidoquinoline-4-yl)-N,N-diethyl-pentane-1,4-diamines (3) with p-chlorophenylisothiocyanate (4b) methylisothiocyanate (4c) ethylisothiocyanate (4d) and tert-butylisothiocyanate (4e) by above mentioned method and enlisted in Table 1.

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