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Synthesis of (2-butyl-4-Substitutedthiamidothiocarbamido-1- {[2-(1h-tetrazol 5-yl) biphenyl- 4-yl] methyl}-1h-imidazol-5-yl) Methanol

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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(2-butyl-4- substitutedthiamidothiocarbamido-1- {[2-(1H-tetrazol-5-yl) biphenyl- 4-yl] methyl}-1H- imidazol-5-yl) methanol(5) by interacting (2-butyl-4-thiocarbamido-1-{[2-(1H-tetrazol-5-yl) biphenyl- 4-yl] methyl}-1H- imidazol-5-yl) methanol(3) with various isothiocynates(4) in ethanol-acetone medium. The(2-butyl-4- thiocarbamido-1-{[2-(1H-tetrazol-5-yl) biphenyl- 4-yl] methyl}-1H- imidazol-5-yl) methanol(3) was synthesized by interacting(2-butyl-4 chloro-1-{[2-(1H-tetrazol-5yl) biphenyl 4yl] methyl}-1H -imidazol-5- yl) methanol (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,(2-butyl-4- thiocarbamido-1-{[2-(1H-tetrazol-5-yl) biphenyl- 4-yl] methyl}-1H- imidazol-5-yl) methanol, acetone and ethanol.

INTRODUCTION

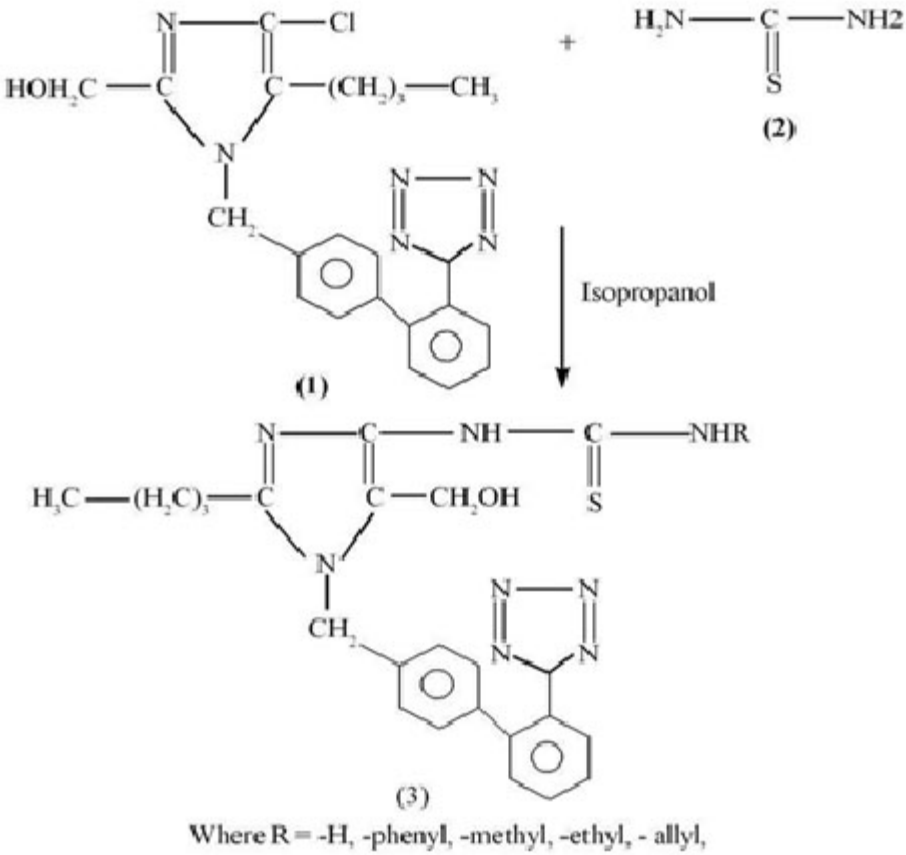
Recently in this laboratory, the synthetic applications of cyanoguanidine had been briefly explored.[1] As evident from the structure of the(2-butyl-4- thiocarbamido-1-{[2-(1H-tetrazol-5-yl) biphenyl- 4-yl] methyl}-1H- imidazol-5-yl) methanol(3), it was observed that there are various reactive sites in this molecule for the reactions. This molecule possesses –thio,-amino, -butyl and -alcoholic 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 thioureas and alkyl or arylisothiocyanates have been investigated in sufficient details [2-7].Some of these compounds showed remarkable pharmaceutical and biological activities[8]. The synthesized heteroacycles are used as a best

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

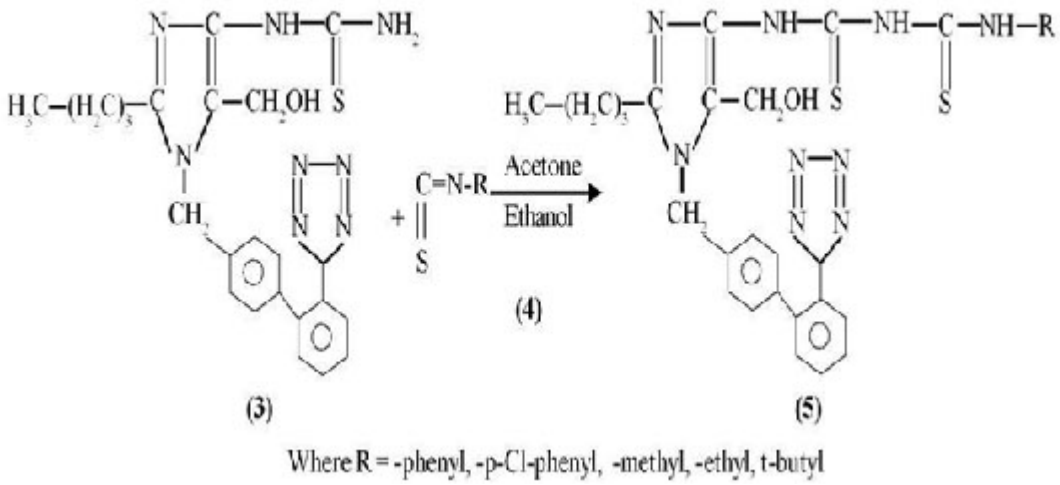
An exhaustive literature survey on substitutedbiureto, 1,2-diazole,1,2,3,4-tetrazoles and benzonido nucleus containing drugs created their own identity in medicinal and pharmaceutical sciences[8-10]. Hence taking all these things into considerations interaction of(2-butyl-4- chloro-1-{[2-(1H-tetrazol-5yl) biphenyl 4yl] methyl}-1H-imidazol-5- yl) methanol (1) with thiourea(2) in isopropanol medium was investigated to synthesize (2-butyl-4- thiocarbamido-1-{[2-(1H-tetrazol-5-yl) biphenyl- 4-yl] methyl}-1H- imidazol-5-yl) methanol(3) (Scheme-1), (2-butyl-4-thiocarbamido-1-{[2-(1H-tetrazol-5-yl) biphenyl- 4-yl] methyl}-1H- imidazol-5-yl) methanol was then interacted with alkyl or aryl isothiocynates (4) in acetone-ethanol medium to isolate yet new series of(2-butyl-4- substitutedthiamidothiocarbamido-1-{[2-(1H-tetrazol-5-yl) biphenyl- 4-yl] methyl}-1H-imidazol-5-yl) methanol(5) (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.



Scheme 1.



Scheme 2.

Table 1.

Sr. No.	2-Butyl-4-substitutedthiamidothiocarbamido -1- {[2-(1H-tetrazol-5-yl) biphenyl- 4-yl] methyl}-1H-imidazol-5-yl) methanol	Yield %	m.p. °C
1	.....-p-Cl-phenyl.....	76	167
2	.....methyl.....	84	148
3	.....ethyl.....	76	192
4	.....tert-butyl.....	89	143

(2-Butyl-4- thiocarbamido-1-{[2-(1H-tetrazol-5-yl) biphenyl- 4-yl] methyl}-1H- imidazol-5-yl) methanol(3))

A mixture of(2-butyl-4- chloro-1-{[2-(1H-tetrazol-5yl) biphenyl 4yl] methyl}-1H- imidazol-5-yl) methanol (1) (0.1M), thiourea (2a) and isopropanol (40ml) was refluxed on boiling water bath for 4 hrs. During boiling suspended(2-butyl-4- chloro-1-{[2-(1H-tetrazol-5yl) biphenyl 4yl] methyl}-1H- imidazol-5- yl) methanol 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 178 °C. (D)

Properties

It is white, crystalline solid having melting point 178^{0}C. (D). It gave positive test for nitrogen and sulphur. Desulphurised with alkaline plumbite solution. It formed picrate, melting point 148° C.

Elemental analysis

C [(found 63.1%) calculated 64.68%], H [(found 5.27%) calculated 5.57%], N [(found 19.21%) calculated 20.81 %], S [(found5.18%) calculated 5.94%]

IR Spectra

The IR spectra was carried out in KBr pellets and the important absorptions can be correlated as, (cm^{-1}) 3184.8 (N-H stretching), 2931.3[C-H(Ar)] stretching,1533.8 (C-N stretching), 1496.3 (=C=NH imino), 1324.5(C-N stretching), 839 (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 ä9.89-9.22 ppm. Ar-CH_2 protons at ä 7.77-7.01 ppm -NH at ä 5.27 ppm. –CH_2 protons at 3.05 ppm. –CH_3 protons at 1.4-1.7ppm.

(2-Butyl-4-phenylthiamidothiocarbamido-1- {[2-(1H-tetrazol-5-yl) biphenyl- 4-yl] methyl}-1H-imidazol-5-yl) methanol(5a)

A mixture of(2-butyl-4- thiocarbamido-1-{[2-(1H-tetrazol-5-yl) biphenyl- 4-yl] methyl}-1H-imidazol-5-yl) methanol(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.188^{0}C.

Examination of Product

It is white crystalline solid having melting point 188^{0}C. It gave positive test for nitrogen and sulphur. Desulphurised with alkaline plumbite solution.

C [(found 59.1%) calculated 60.30%], H [(found 5.17%) calculated 5.02%], N [(found 20.21%) calculated 21.10 %], S [(found10.18%) calculated 10.72%]

IR Spectra

The IR spectra was carried out in KBr pellets and the important absorptions can be correlated as, (cm^{-1}) 3393.6 (N-H stretching),2362.7 [C-H(Ar)] stretching, 1661.6 (C-N stretching), 1101.6 (=C=NH imino), 517.3 (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 ä7.941-8.54 ppm. Ar-CH_2 protons at ä 6.85 ppm, -NH at ä 3.97 ppm. –CH_2 protons at 2.12-2.86 ppm. –CH_3 protons at 1.27 ppm Similarly,(2-butyl-4-p-Cl-phenylthiamido-thiocarbamido-1- {[2-(1H-tetrazol-5-yl) biphenyl-4-yl]methyl}-1H-imidazol-5-yl)methanol(5b),2-butyl-4-methythiamidothiocarbamido-1- {[2-(1H-tetrazol-5-yl) biphenyl- 4-yl] methyl}-1H imidazol-5-yl) methanol(5c),,(2-butyl-4-p-Cl-ethylthiamido-thiocarbamido-1-{[2-(1H-tetrazol-5-yl)biphenyl-4-yl] methyl}-1H- imidazol-5-yl) methanol(5d) and (2-butyl-4-tert-butylthiamidothiocarbamido-1- {[2-(1H-tetrazol-5-yl) biphenyl- 4-yl] methyl}-1H- imidazol-5-yl) methanol(5e)were synthesized by interacting (2-butyl-4- thiocarbamido-1-{[2-(1H-tetrazol-5-yl) biphenyl- 4-yl] methyl}-1H- imidazol-5-yl) methanol(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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