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Original Article

Synthesis and Structural Elucidation of A New Series of 1-Formamidino-(N-Substitutedthioamido)-5-Substituted -2-THIO-4-Imino Biurets

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ABSTRACT

Intraction of phenylthiocarbamide were carried out with dicyanamide to obtain N"-phenylformamidinoformamidinothiocarbamide(1b). which were further reacted with different alkyl/arylisothiocyanate in 1:1 molar ratio in acetone medium to obtain series of 1-formamidino-(n-substitutedthioamido)-5-substituted -2-thio-4-iminobiuret. (2) which may posses different pharmaceutical, industrial, medicinal, agricultural important which may be further cyclise to synthesis yet new series of 5 and 6 membered hetercyclic compounds. The structure of all these compounds were established on the basis of elemental analysis, IR and PMR spectral data. Rotective agent in the treatment of parkinsons disease along with standard anti-Parkinson agents.

Keywords: Dicyandiamide, 1, 3-Diformamidinothiocarbamide, Cyanoamidinothiocaramides, synthesis.

INTRODUCTION

Thiocarbamides and their derivatives possess various pharmaceutical, agricultural, medicinal and values[1-4]. Intraction of different thiocarbamides and substituted cyanamides were carried out to synthesised respective substituted thiocarbamides, amidinothiocarbamides (salt) and their derivatives.[5-8] Amidinothiocarbamides and their derivatives were successfully cyclised into respective thiadiazines and s-triazines by using different reagent and different reaction conditions. [9-21] Dicyanamide has interesting to carry out the reaction with thiourea in presence of dil. hydrochloric acid in acetone medium to obtain 1,3-

diformamidinothiocarbamide, having two amino groups at the terminal ends.

EXPERIMENTAL

All chemicals used were of analar grade. Substituted isothiocyanates were prepared according to literature method.[22] Melting point of all synthesised compounds were determine in open capillary and uncorrected, IR spectra were recorded on Perkin-Elmer spectrometer in the range 4000-400^{cm-1} in Nujol mull as KBr pellets.PMR spectra were recorded with TMS as internal standard using $CDCl_3$ and DMSO-d_6. The purity of the compounds were checked on silica gel-G plates by TLC with layer thickness of 0.3 mm. All compounds gave satisfactory C, H, N and S elemental analysis.

RESULT AND DISCUSSION

N-phenylformamidinoformamidinothiocarbamide (1b)

A mixture of dicyanamide, phenylthiourea, conc. hydrochloric acid, distilled water was refluxed in acetone medium for 8 h. on water bath. The precipitated solid was collected by filtration and recrystalised from aqueous ethanol Yield 61%. m.p. 182^{0}C. IR spectra of compound shows ν(N-H) 3373.9cm^{-1}, ν(C-H)(Ar) 3150.0cm^{-1}, ν (C=N) 1669.0cm^{-1}, ν(C-N) 1394.7 cm^{-1}, ν(C=S) grouping 1181.3 cm^{-1}, ν(C=NH) grouping 1527.4cm^{-1}. The PMR spectra of compounds showed signals due to N-H protons at δ 3.9-4.3 ppm Ar-NH protons at δ 6.3 ppm, Ar-H protons at δ 5.1-5.9 ppm and the signal at δ 3.1-3.2 ppm is due to moisture in DMSO-d_6. and δ1.3-2.3 ppm is due to DMSO. Found (Calcd.) C = 15.29% (15.51%) H = 3.98% (4.31%) N = 33.13% (36.20%) S = 13.69% (13.79%).

1-formamidino-(n-phenylthioamido)-5-phenyl -2-thio-4-iminobiuret. (2a).

Mixture of (1b) (0.05mol) phenylisothiocyanate (0.05 mol) and acetone (50ml) was refluxed for 12 h. on water bath in 1:1 molar ratio. The mixture was filtered and filtrate during distillation yielded the crystals of 4a.Yield (79%). m.p. 245^{0}C ; IR spectra of compound shows ν(N-H) 34.53.9cm^{-1}, ν(C-H)(Ar) 3110.5cm^{-1}, ν(C=N) 1576.2cm^{-1}, ν(C-N) 1296.6cm^{-1}, ν(C=S) grouping 1135.3 cm^{-1}, ν(C=NH), grouping 1627.8cm^{-1}. The PMR spectra of compounds showed signals due to N-H protons at δ 3.4-4.7 ppm Ar-NH protons at δ 6.7 ppm, Ar-H protons at δ 5.7-6.3 and the signal at δ 3.5-3.7 ppm is due to moisture in DMSO-d_6. and δ1.5-2.1 ppm is due to DMSO. Found (Calc.) C = 51.79% (51.75%) H = 4.42% (4.58%) N = 26.31% (26.41%) S = 17.22% (17.25%).

Similarly others compounds (2b-2f) were synthesised by above mention method and enlisted in table-I

CONCLUSION

As outline in synthesis process, important novel iminobiuret have been synthesized. All the structure of the above compounds was in good agreement with Spectral and Analytical data. and also shows novel biological activity.

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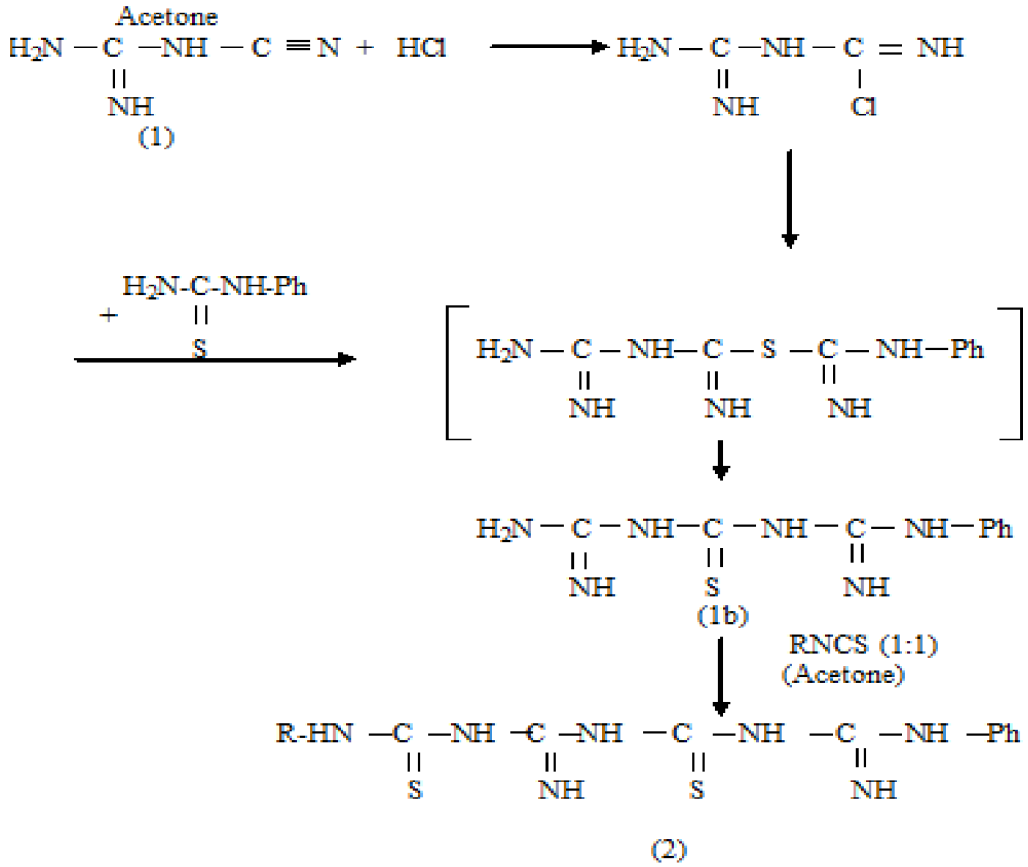
Table 1: Physical data and analysis of the compounds (2a-f)

Compd.	R	m.f.	Yield	m.p. (^{0}C)
2a	phenyl	$C_{16}H_{17}N_7S_2$	79	245
2b	*p*-chlorophenyl	$C_{16}H_{16}N_7S_2Cl$	72	254
2c	*p*-tolyl	$C_{17}H_{19}N_7S_2$	79	267
2d	ethyl	$C_{12}H_{17}N_7S_2$	68	238
2e	methyl	$C_{11}H_{15}N_7S_2$	73	234
2f	*t*-butyl	$C_{14}H_{21}N_7S_2$	62	261



Scheme

Where R = **2a** phenyl ; **2b** *p*-chlorophenyl; **2c** *p*-tolyl; **2d** ethyl; **2e** methyl; **2f** *t*-butyl