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A NOVEL SYNTHESIS OF [(4-AMINO)-PHENYL] SUBSTITUTED THIOCARBAMIDES

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Abstract-

Recently in this laboratory the synthesis of [(4-amino)-phenyl]substituted thiocarbamides (3a-b) were carried out by the interactions of 4-chloroaniline(1) with various thiourea (2a-b) in 1:1 proportions in isopropanol medium for 4 hours. The synthesized compounds were characterized on the basis of elemental analysis, chemical characteristics and spectral data.

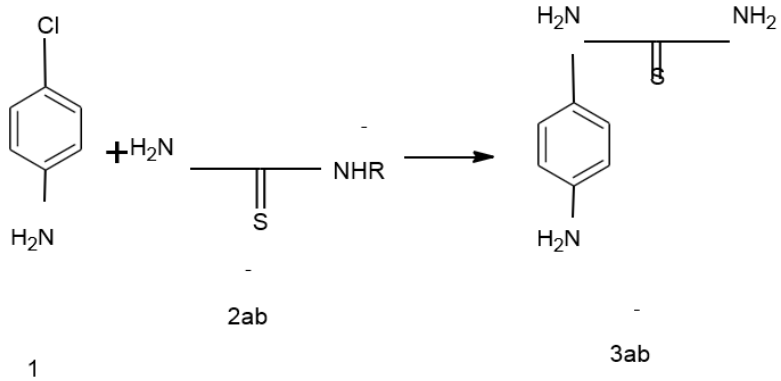
Keywords-4-chloroaniline, thiourea, phenylthiourea, isopropanol.

Introduction-

The literature survey of 4-chloroaniline is use as pesticides, bacteriostatic, dyes, drugs and disinfectant agent. The literature survey of thiocarbamide also shows that the thiocarbamido nucleus containing drug have their own importance in pharmaceutical chemistry because thus drug show antiemetic, analgesic, anti-emetic anti-pyretic, anti-ulcer, anti-malarial properties[1-4].

The clinical uses, adverse effect, physiochemical properties and the chemistry of 4-chloroaniline were studied in sufficient details. Thiocarbamide nucleus containing molecules shows various medicinal and pharmaceutical applications.

As wider program of this laboratory in the synthesis of nitrogen and sulphur containing heterocycles and their cyclisation into 5,6, and 7 membered heterocyclic and to investigate their medicinal, pharmaceutical parameters, it was thought interesting to carry out the interactions of 4-chloroaniline with different thiocarbamides in isopropanol medium to isolate a new series of heterocyclic drugs havingbenzene and thiocarbamide nucleus in the same drug. This may enhance the potency of drugand may also introduce new type of drug activity. This type of reaction is heither to unknown. This synthetic approach will be come a milestone and open a new path in pharmaceutical, biochemical,
Medicinal and drug chemistry[4].
Taking all these things into consideration the interactions of 4-chloroaniline(1) and various thiocarbamide (2a-b) were carried out in isopropanol medium. (Scheme-I)



1 2ab 3ab

RESULT AND DISCUSSION

Synthesis of [(4-amino)-phenyl]-1-thiocarbamide(3a)–

A mixture 4-chloroaniline (1) and thiocarbamide (2a) in 1:1 proportions in isopropanol medium were refluxed for 4 hours on water bath. During boiling 4- chloroaniline and thiocarbamide went into the solution and the new product was formed i.e. [(4-amino)-phenyl]-1-thiocarbamide (3a) to be gradually separated out

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which on basification with dilute ammonium hydroxide afforded crystals. It was filtered in hot condition and crystallized with aqueous ethanol to obtain yield 89% and melting point 135^{0}C.

Propertiesof3a–

The compound is light yellow and crystalline in nature and having melting 135^{0}C. It contains nitrogen and sulphur. Desulphurised with alkaline plumbite solution.

Element alanalysis-

Found (%):-Carbon 47.43, Hydrogen 4.74, Nitrogen 27.54 and Sulphur17.57.

Calculated (%):-Carbon 48.56, Hydrogen 5.20, Nitrogen 27.54 and Sulphur 18.49.

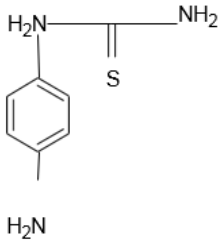
It formed picrate having melting point165^{0}C. From analytical data the molecular formula found to be $C_7H_9N_3S$.

IRSpectra[1-6]:-The IR spectra was carried out in KBr pellets and reproduced on IR plate number MI-01.The important absorption can as follow-

Absorption observed	Assignment (cm^{-1})	Absorption expected (cm^{-1})
3195.52	N-H stretching Amine N-	3400–3000
3391.00	H stretching Imine	3400–3310
	[C=C]stretching benzene C-H	
1621.24	Stretching in benzene	1630-1400
	-C=S	
2907.81	-N-C-N Stretching	3050-2900
1175.66		1225-1025
		1100-1050
1101.40		

NMR Spectra:- The spectrum was carried out in DMSO. This spectrum distinctly displayed the signals due toAr-H protons at δ 6.5612to7.1594ppm, -NH_2 protons at δ 3.4571, =NH at δ 5.1859 ppm.

From the above properties and the spectral analysis the compound (**3a**) was assigned the structure as [(4-Amino)phenyl]-thiocarbamide.



Synthesisof[(4-Amino)phenyl]-3-phenylthiocarbamide–

Amixture 4-chloroaniline (1) and phenylthiocarbamide (2b) in 1:1proportions in isopropanol medium were refluxedfor4 hours on water bath. During refluxing4- chloroaniline and phenylthiocarbamide went into the solution and the new product was formed i.e. [(4-Amino)phenyl]-3-phenylthiocarbamide to be gradually separated out which on basification with dilute ammonium hydroxide afforded crystals. It was filtered in hot condition and crystallized with aqueous ethanol to obtain yield 80% and melting point 110^{0}C.

Propertiesof3b–

The compound is cream and crystalline in nature and having melting 110^{0}C. It contains nitrogen and sulphur. Desulphurised with alkaline plumbite solution.

Elementalanalysis-

Found (%):- Carbon 64.02, Hydrogen 3.28, Nitrogen 17.50 and Sulphur 12.41. Calculated (%):-Carbon65.00,Hydrogen4.16,Nitrogen17.50andSulphur 13.33.

From analytical data the molecular formula found to be $C_{13}H_{13}N_3S$.

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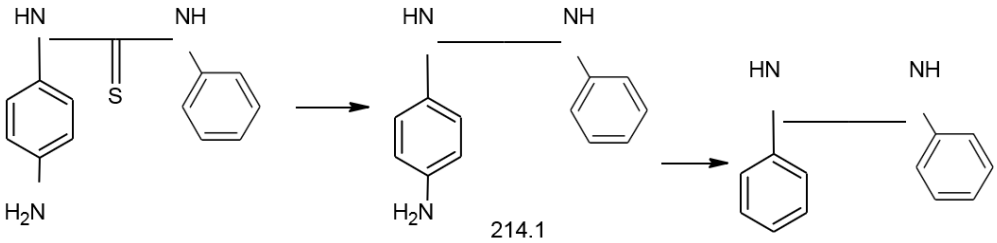
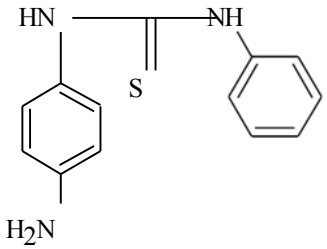
IR Spectra[1-6]:- The IR spectra was carried out in KBr pellets and reproduced on IR plate number MI-2.The important absorption can as follow-

Absorptionobserved	Assignment (cm^{-1})	Absorption expected(cm^{-1})
3415.12	N-H stretching Imine [C=C] stretching benzene C-H	3450–3310
1528.65	Stretchinginbenzene	1630-1400
3011.01	-C=S	3050-2900
1286.58	-N-C-NStretching	1275-1025
1083.08		1100-1050

1. It forms picrate havin gmelting point 169^{0}C.
2. **NMR Spectra**:- The spectrum was carried out in CDCl3. This spectrum distinctly displayed the signals due to Ar-H protons at δ 6.5554 to 7.4395 ppm, -NH2 protons at δ 3.4161 ppm, =NH at δ 9.7926 ppm.
3. **Mass spectrum**:- The Mass analysis of the compound was carried out and reproduced on **Mass Plate No. MI-2**. The fragmentation occurs during the analysis is given in **Mass Scheme-I**



From the above properties and the spectral analysis the compound (**3b**) was assigned the structure as [(4-Amino)phenyl]-3-phenylthiocarbamide



EXPERIMENTAL

The melting point of all the synthesized compounds was recorded using hot paraffin bath. IR spectra were recorded on Shemadzu spectrometer in the range 4000-400 cm^{-1} in KBr pellet's. PMR spectra were recorded Bruker AC - 500F spectrometer with TMS as internal standard using CDCl3 and DMSO as solvent. The purity of compounds was checked on Silica-gel-g plates by TLC within the layer thickness of 0.3 mm. All used were of AR Grade.

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