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Synthesis of 1-Phenylthiocarbamido-5-Formamidino – Substitutedthiocarbamido -2-Imino-4-Thiobiurates by Microwave Technique

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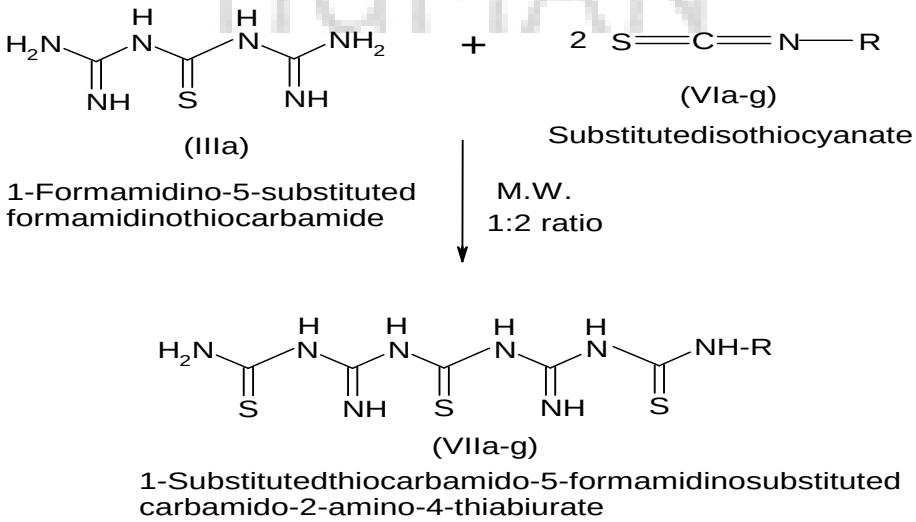
ABSTRACT

"Non-conventional" synthetic method has shown broad applications as a very efficient way to accelerate the course of many organic reactions, producing high yields, higher selectivity and lower quantities of side products consequently easier work-up and purification of the products. Synthesis of a novel series of 1-substitutedthiocarbamido-5-formamidino-substitutedthiocarbamido-2-imino-4-thiobiurates **(VIIa-g)** having high product yield by the interactions of 1-formamidino-5-formamidinothiocarbamide **(IIIa)** and various isothiocyanates **(VIa-g)** by microwave technique. The green chemistry parameters were maintained and perfidious byproducts were avoided. The synthesized compounds were recrystallized and the structure of synthesized compounds were justified and established on the basis of elemental analysis, chemical characteristics and spectral studies.

NTRODUCTION

The thiocarbamido, amidinothiocarbamido and thioamido nucleus containing heteroacycles and heterocycles have their own identity and importance in pharmaceutical, medicinal, agricultural and industrial sciences[1-4]. It is also renoved that thiocarbamides, bis-formamidinosulphides, amidinothiocarbamides containing nucleus which act as a drug which enhances the potency and therapeutic value[5-11]. N-Aryl/alkylisocyanodichloride was prepared according to literature method[12]. The evidence of interaction of 1,3-diformamidinothiocarbamides with alkyl/arylisothiocyanate in 1:2 molar ratio in microwave oven to obtain yet new series of 1-substitutedthiocarbamido-5-formamidinosubstitutedthiocarbamido-2-imino-4-thiobiurates respectively which is hitherto unknown. These molecules possess various medicinal, agricultural, industrial and biochemical applications and importance; hence the present research scheme was designed to describe somewhat suitable and direct method for a synthesis of the novel series of 1-substitutedthiocarbamido-5-formamidinosubstitutedthiocarbamido-2-imino-4-thiobiurates.

Therefore, it is quite interesting to investigate the interactions of 1-formamidino-5-formamidinothiocarbamide **(IIIa)** and various isothiocyanates **(VIa-g)** in 1:2 molar proportions in microwave irradiation technique to synthesize a novel series of 1-substitutedthiocarbamido-5-formamidinosubstitutedthiocarbamido-2-imino-4-thiobiurates **(VIIa-g)**. The tentative reaction for the formation of product is depicted below, (**Scheme-III**).



Scheme-III

Experimental:

Specific rotations were measured on Equiptronics Digital Polarimeter at 28^0 C in $CHCl_3$. IR spectra were recorded on Perkin-Elmer spectrum RXI FTIR spectrophotometer (4000-450 cm^{-1}). ^1H NMR was recorded in $CDCl_3$ on Bruker DRX-300 spectrometer operating at 300 MHz. The mass spectra were recorded on Jeol-SX-102(FAB) instrument.

Synthesis of 1-phenylthiocarbamido-5-formamidinophenylthiocarbamido-2-imino-4-thiobiurate (VIIb)

A mixture of 1-formamidino-5-formamidinothiocarbamide (0.1m) **(IIIa),** phenylisothiocyanate (0.2m) **(VIb)** was kept in microwave oven for 2 minutes. Then the reaction mixture was poured into ice cubes with vigorous stirring, ivory crystals were obtained. These crystals were washed several times with ether and recrystallised from aqueous ethanol. (Yield 96%, melting point 168^0C).

Properties: It is yellow colored crystalline solid having melting point 147^0C.

It gave positive test for nitrogen and sulphur. It was desulphurised by alkaline plumbite solution which clearly indicates the presence of C=S group. It also gave positive test for imino group. It was soluble in water, ethanol, DMSO-d_6 while insoluble in carbon tetrachloride, chloroform, benzene, dioxane and petroleum ether. It formed picrate having melting point 160^0C. The R_f value was found to be 0.37.

Elemental Analysis: C [(found 46.41%) calculated 47.44], H [(found 03.21%) calculated 04.18], N [(found 26.04%) calculated 26.04], S [(found 21.30%) calculated 22.32].

IR spectrum (cm^{-1}): The IR spectrum was carried out in KBr-pellets: 3377.10 (N-H stretching), 2817.60 (Ar-CH stretching), 2192.40(S=C=N streching), 1662.10 (C=S stretching), 1627.10 (C=NH imino group), 1472.03 (N-C=S stretching), 1194.50 (C-N stretching). **NMR Spectrum:** The NMR spectrum of compound was carried out in $CDCl_3$ and DMSO-d_6**.** This spectrum distinctly displayed the signals due to -NH proton at δ3.5431 ppm, CH_2 protons at δ 2.5763 ppm, CH_3 protons at δ 2.5763 ppm,

Similarly, 1-methylthiocarbamido-5-formamidinomethylthiocarbamido-2-imino-4-thiobiurate **(VIIc),** 1-(p-chlorophenyl)thiocarbamido-5-formamidino (p-chlorophenyl)thiocarbamido-2-imino-4-thiobiurate **(VIId),** 1-o-tolylthiocarba mido-5-formamidino(o-tolyl)thiocarbamido-2-imino-4-thiobiurate **(VIIe),** 1-(m-tolyl)thiocarbamido-5-formamidino(m-tolyl)thiocarbamido-2-imino-4-thiobiurate **(VIIf),** 1-(p-tolyl)thiocarbamido-5-formamidino(p-tolyl)thiocarbamido-2-imino-4-thiobiurate **(VIIg)** were synthesized by the interactions of 1-formamidino-5-formamidinothiocarbamide **(IIIa)** with various methylisothiocyanate **(VIc),** p-chlorophenylisothiocyanate **(VId),** o-tolylisothiocyanate **(VIe),** m-tolylisothio-cyanate **(VIf),** p-tolylisothiocyanate **(VIg)** respectively by the above mentioned method in **Experiment No.** 1,2 and 4 and enlisted in **Table No. 1.**

Table No. 1

Sr. No.	**1-Substitutedthiocarbamido-5-formamidinosubstitutedthiocarbamido-2-imino-4-thiobiurate**	**Yield (%)**	**M.P. ^{0}C**
1.	1-**Methyl**thiocarbamido-5-formamidino**methyl**thiocarbamido-2-imino-4-thiobiurate	95	165
2.	1-**(p-Chlorophenyl)**thiocarbamido-5-formamidino**(p-chlorophenyl)**thiocarbamido-2-imino-4-thiobiurate	88	175
3.	1-**(o-Tolyl)**thiocarbamido-5-formamidino**(o-tolyl)**thiocarbamido-2-imino-4-thiobiurate	96	185
4	1-**(m-Tolyl)**thiocarbamido-5-formamidino**(m-tolyl)**thiocarbamido-2-imino-4-thiobiurate	86	124
5	1-**(p-Tolyl)**thiocarbamido-5-formamidino**(p-tolyl)**thiocarbamido-2-imino-4-thiobiurate	98	161

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