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Synthesis Of 1-[S-TAG-N Substituted Phenylthioamido] Dicyandiamide By Reflux Method And Its Characterization.

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ABSTRACT: In a view of medicinal, pharmaceutical, agricultural and industrial importance recently in our laboratory a novel series of 1-[S-TAG-N substituted thioamido] dicyandiamide have been synthesized successfully by refluxing TAG Br with cyanoamidino substituted thio-carbamides and 1-formamidino-3-substituted formidino-thiocarbamides in isopropanol for 3hrs. The justifications of the structure of these newly synthesized compounds have been established on the basis of chemical characteristics, elemental analysis and IR, PMR and mass spectral analysis. These materials found several applications in pharmaceutical industry and also in medicinal chemistry.

KEYWORDS: Reflux Synthesis, pharmaceutical applications, NMR.

I. INTRODUCTION

Glucosyl group or its derivatives when attached to the sulphur of the sulphur containing hetreroacycles and heterocycles are commonly referred as "Thioglucosides." These compounds have their own identity and remarkable significance in various industries, and also in pharmaceutical, medicinal, biochemical and organic chemistry. The possess anti-analgesic, anti-diabetic, anti-pyretic, anti-tubercular properties. As evident from the structure of cyanoamidino substituted thiocarbamide, it was observed that there are various reactive sites in this molecule for the reactions. This molecule possesses –SH, -CN, -NH_2 important reactive sites for the reactions. As a wider program of this laboratory in the synthesis of nitrogen, nitrogen and sulphur containing heteroacycles and heterocycles. The interactions of dicyandiamide with various thioureas and alkyl/arylisothiocycnates had been investigated in sufficient details in various reaction conditions [1-4]. Some of these compounds showed noticeable pharmaceutical and biological values [5-6]. These heteroacycles were also classified in 5 and 6 membered heterocycles viz. thiadiazoles, dithiazoles, hectors bases, thiadiazines and triazines. These heterocycles posses their own identity and significance in pharmaceutical, medicinal, agricultural, industrial and biotechnical sciences [7-10]. S-glucosides and N-glucosides had been found several applications in industry and also in medicinal chemistry [11-12].

An exhaustive literature survey about tetra-O-acetyl-B-D-glucopyranosylbromide and tetra-O-benzoyl-B-D-glucopyrasonylbromide showed that these two analog play the great role in the synthesis of S and N-glucosylated heteroacycles and heterocycles. Very few thioglucosides of thiocarbamide were reported earlier [15]. Hence, it was thought interesting to synthesize1-[S-TAG-N substituted thioamido] dicyandiamide. With this aim and objectives the interactions of tetra-O-acetyl-b-D-glucopyranosylbromide with cynoamidinosubstitutedthiocarbamides and 1-formamidino-3-substituted formamidinothiocarbamide in isopropanol medium were investigated to isolate 1-[S-TAG-N substituted thioamido] dicyandiamide.

II. EXPERIMENTAL

1-[S-TAG-N-phenylthioamido] dicyandiamide was synthesized by refluxing a mixture of tetra-O-acetyl-b-D-glucopyranosylbromide and cyanoamidino phenylthiocarbamide in isopropanol medium, on water bath for 3 hrs. During refluxing the suspended tetra-O-acetyl-b-D-glucopyranosylbromide and cyanoamidinophenylthiocarbamide

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went into the solution and clear solution was obtained. It was kept for 15 Hrs. at room condition. It was then mixed with distilled water, small quantity of semisolid was obtained, it was filtered. The aqueous solution on basification with dilute ammonium hydroxide, afforded dark brown colored sticky solid which on tritation several times with petroleum ether gave brown crystals. It was re-crystallized with aqueous ethanol, yield-78%, m. p. 167°C. The probable reaction mechanism of the formation of is as depicted below.

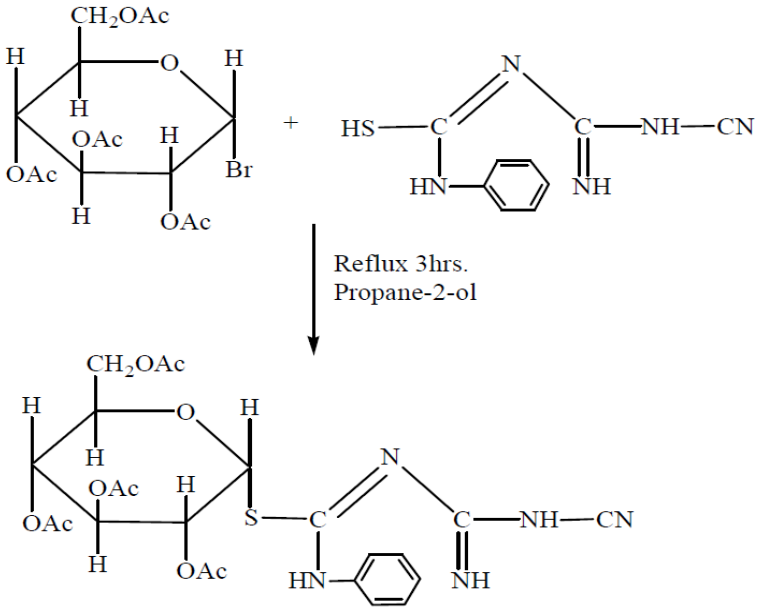

Figure 1 Reaction mechanism of 1[S-TAG- N-phenylthioamido]dicyandiamide

III. RESULTS AND DISCUSSIONS

The IR spectrum of compound 1-[S-TAG-N-phenylthioamido] dicyandiamide was carried out in KBr pellets and is reproduce on plate No. IR-2.1. The IR spectrum clearly indicated the bands due to ν-NH, ν-C-H(Ar), ν-C=O, ν-C=N, ν-RC-N, ν-RC-S and an important absorption can be correlated in table no. 1.

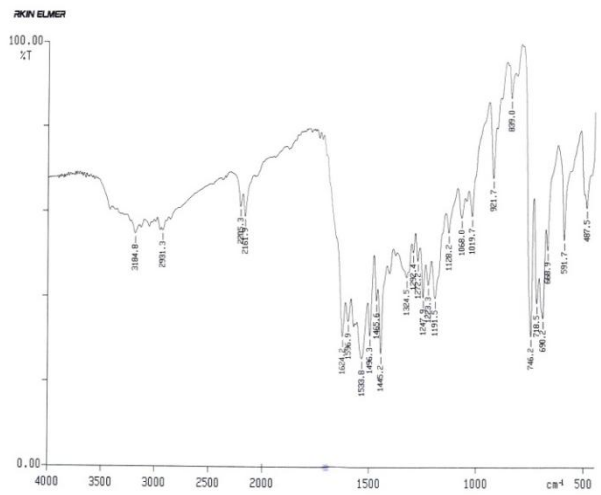

Figure 2 FT-IR Spectrum of 1[S-TAG- N-phenylthioamido]dicyandiamide

Absorption Observed	Assignment	Absorption expected (cm^{-1})
3184.8	N-H Stretching	3500-3000[16-19]
2931.3	C-H (Ar) Stretching	3150-2900[20]
1533.8	C=NH stretching	1789-1478[21]
1496.3	C=N stretching	1789-1471[21]
1324.5	C-N stretching	1340-1250[22]
839.0	glucopyranosyl C-H deformation	844±8[23]
746.2	C-S stretching	800-600[24]

Table 1 Major absorption peaks in FT-IR Spectrum of 1[S-TAG- N-phenylthioamido]dicyandiamide

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The PMR spectrum of compound 1-[S-TAG-N-phenylthioamido] dicyandiamide was carried out in DMSO-d_6 and $CDCl_3$ and shown in figure 3. This spectrum distinctly displayed the signals due to –NH protons at δ 9.89 - 9.22 ppm, Ar. Protons at δ 7.77 - 7.01 ppm, the signals at δ 6.99 – 6.27 ppm are due to protons of pyranosyl ring, signals due to ester group proton at δ 3.05 ppm and signals due to proton of Kriz -139 p. R_3CH- at δ 1.4-1.7 ppm.

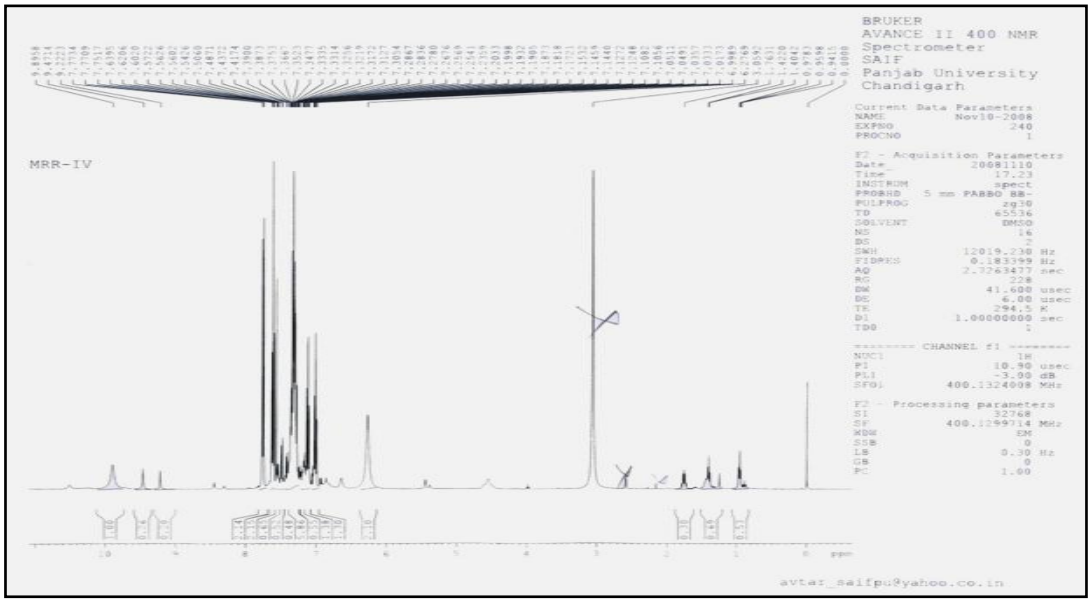

Figure 3: PMR spectra of 1[S-TAG- N-phenylthioamido]dicyandiamide

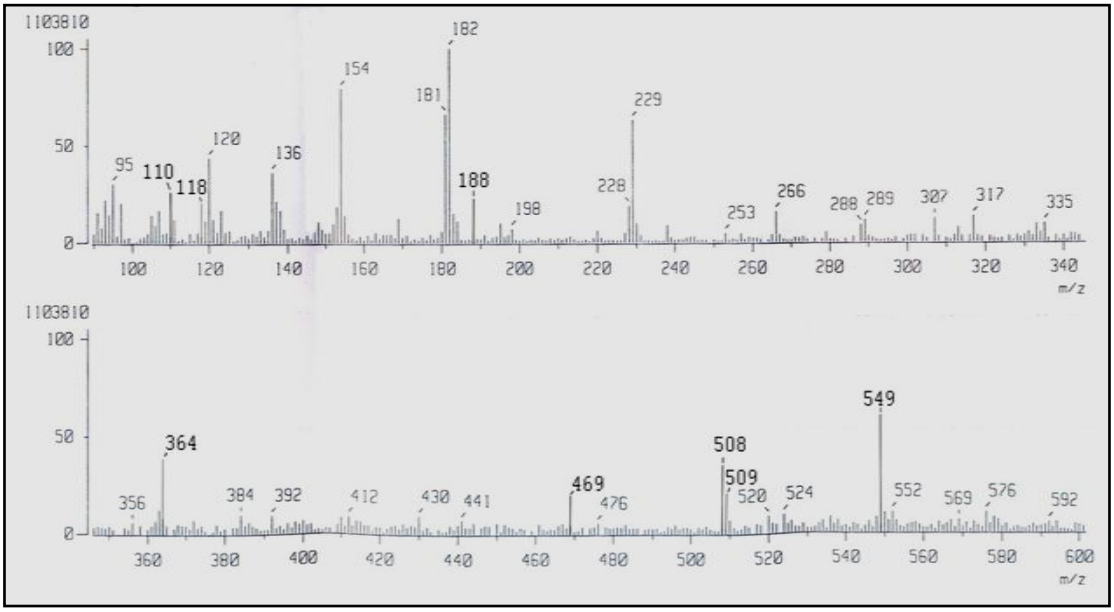

Figure 4 Mass Spectrogaph of 1[S-TAG- N-phenylthioamido]dicyandiamide

The FAB mass spectrum of 1-[S-TAG-N-phenylthioamido] dicyandiamide shown in figure 4, was recorded at room temperature by using meta nitrobenzyl alcohol as the matrix m^+ peak as well as other fragment peaks and the probable fragmentation pattern of the molecular ion. While the mass spectrum is reproduced on plate No. Mass – 2.1 From the

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above properties and spectral analysis the compound was assigned, the structure as 1-[S-TAG-N-phenylthioamido] dicyandiamide.

IV. CONCLUSIONS

The compound 1-[S-TAG-N-phenylthioamido] dicyandiamide was successfully prepared which grown brown crystalline solid with m. p. 167°C. The synthesized sample was characterized by FT-IR, PMR and mass spectrograph indicating formation of the desired product. The compounds were studied for their antimicrobial activity and all the pathogen tested during analysis. From the results it was clear that compound showed remarkable and considerable antimicrobial activity against organism. The activity of compounds were tested against all the pathogen by disc diffusion method. The antimicrobial activity of different compound against micro-organisms were examined in the presence study and their potency, were assessed by the presence or absence inhibition zone and zone diameter.

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