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NOTE

Synthesis and Structural Elucidation of Substituted s-Triazines

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2-Amino-4-hydroxy-s-triazine was synthesized by the interaction of cyanoguanidine with formic acid. On further reaction with various isothiocyanate in acetone-ethanol medium to produce 1-(4-hydroxy)-s-triazino-3-substituted thiocarbamide (**III**). The products synthesized in these reactions were characterized on the basis of conventional elemental analysis, chemical characteristics and through IR and NMR spectral analysis.

Key Words: s-Triazines, Guanidine, Isothiocyanates, Substituted thiocarbamide.

Heterocyclic chemistry is the chemistry of cyclic compounds having hetero atoms in ring like nitrogen, sulphur and oxygen along with carbon and other elements. Heterocyclic compounds having s-triazino nucleus have their own importance in medicinal, agricultural, pharmaceutical and industrial fields[1-3]. In present studies it was proposed to carry out the interactions of cyanoguanidine with formic acid to synthesize s-triazine and to investigate their reactions with substituted isothiocyanates in different reaction mediums. Out of which acetone-ethanol medium shows better yield and purity of compound which is described to produce 1-(s-triazino)-3-substituted thiocarbamide[4] in order to synthesize more active drugs.

All the chemicals used were of AnalaR grade alkyl/arylisothiocyanates were prepared according to literature method and melting points of all synthesized compounds were determined in open capillary and are uncorrected. IR spectra were recorded on Perkin-Elmer spectrophotometer in the range 4000-400 cm^{-1} in KBr pellets. PMR spectra were recorded with TMS as an internal standard using $CDCl_3$ and DMSO-d_6. The purity of the compounds was checked on silica gel-G plates by TLC.

Synthesis of 2-amino-4-hydroxy-s-triazine (I)[5,6]: When interaction of cyanoguanidine has been carried out with 85 % formic acid as described by earlier workers on an oil bath at temperature 120-30 °C for 6 h, initially the vigorous reaction was set in and after some time a clear solution was obtained. This on

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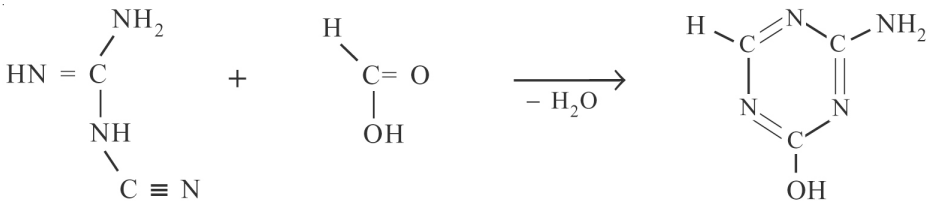
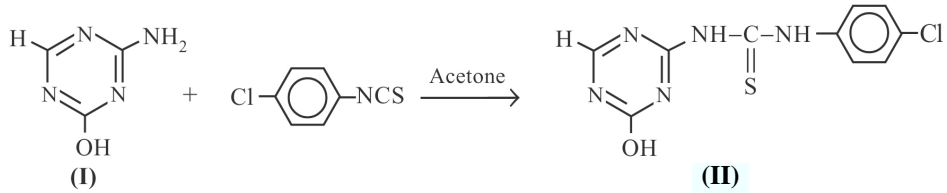
cooling afforded a solid which was triturated several times with ethanol, gave a product, m.p. 175-80 °C, (yield: 7.2 g, 86 %), soluble in cold water, aqueous sodium hydroxide, dilute sulphuric acid, hydrochloric acid and nitric acid and was sparingly soluble in hot ethanol and was completely insoluble in acetone and benzene. It formed picrate (m.p. 193-93 °C). Found. (%) C 31.25, H 2.97, N 48.52 ($C_3H_4N_4O$) requires C 32.14, H 3.57, N 50.00. IR (KBr, ν_{max}, cm^{-1}): 731.0 (iso form of s-triazino ring), 1339 (Ar-O), 1582 (N-C=N), 1660 (>C=N), 3259 (N-H). PMR (δ, $CDCl_3$ + DMSO-d_6) 9.30 (Ar-OH), 8.83 (triazino-H), 7.2-7.6 (Ar-NH), 3.97 (DMSO).



Cyanoguanidine Formic acid **(I)** 2-Amino-4-hydroxy-s-triazine

Synthesis of 1-(4-hydroxy)-s-triazino-3-*p*-chloro-phenylthiocarbamide (II)[7]: Condenzation of 2-amino-4-hydroxy-s-triazine (**I**) was carried out with *p*-clphenyl-isothiocyanate in acetone-ethanol medium to obtain yellowish needle shaped crystals of (**II**), m.p. 207 °C, (yield: 4.3 g, 79 %), sparingly soluble in hot water, ethanol, acetone and benzene and soluble in mineral acids, acetic acid and dioxane. Desulfurized with alkaline plumbite solution. The R_f value was found to be 0.3 for dioxane as a solvent on silica gel-G plate with layer thickness of 0.4 mm. Found (%) C 42.38, H 2.58, N 24.82, S 11.12, Cl 11.88 ($C_{11}H_{10}N_5OSCl$) requires C 42.62, H 2.84, N 24.86, S 11.36, Cl 12.61. IR (KBr, ν_{max}, cm^{-1}): 607.0 (C-S), 772.6 (iso form of s-triazino ring), 1277 (C-O), 1359 (C=S), 1497 (>C=N), 1661 (N-C=N), 2945 (intermolecular hydrogen bondeding in OH), 3334.0 (N-H). PMR (δ, $CDCl_3$ + DMSO-d_6) 8.05-8.35 (triazino-OH), 7.81 (Ar-H), 6.62 (triazino-H), 5.37 (Ar-NH), 3.47-3.62 (triazino-NH), 2.59 (DMSO).



(I) 2-Amino-4-hydroxy-s-triazine **(II)** 1-(4-Hydroxy-s-triazino)-3-*p*-chloro thiocarbamide

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