Asian Journal of Chemistry Vol. 18, No. 4 (2006), 3137–3138

NOTE

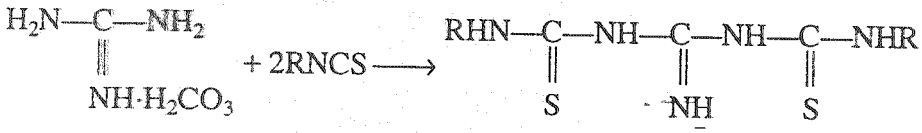
Synthesis of 1,3-bis (N-Substituted Thioamido) Guanidine

D.T. TAYADE† and M.E. SHELKE*
Department of Chemistry, Jijamata Mahavidyalaya, Buldhana, India
E-mail: meshelke@rediffmail.com

In the present note, an investigation of the reaction of guanidine carbonate with aryl/alkyl isothiocyanate to synthesize new series of 1,3-bis(N-substituted thioamido) guanidine is reported.

Key Words: Synthesis, Guanidine.

Guanidine salts have been invariably used in the synthesis of many 6-membered nitrogen containing compounds, particularly thiadiazine, pyrimidine and *s*-triazine derivatives containing nitrogen and sulphur heterocycles, which have their own identity and importance in many fields. So it was thought interesting to interact guanidine carbonate (1) with aryl/alkylisothiocyanate (2) in 1 : 2 molar ratio to isolate 1,3-bis(N-substituted thioamido) guanidine (3) which are hereto not known (**Scheme-1**). These compounds will open new series of heterocyclic compounds, which will be cyclized with different reactants under different reaction conditions to obtain thiadiazolidines, thiadiazines, pyrimidines and *s*-triazines.



where, R = **3a** (phenyl), **3b** (*p*-chlorophenyl), **3c** (*p*-tolyl), **3d** (ethyl), **3e** (methyl), **3f** (*t*-butyl).

Scheme-1

All chemical used were of AnalaR grade. Substituted isothiocyanate were prepared according to literature method[1]. Melting points of all synthesized compounds were determined in open capillary and are 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. TLC checked the purity of the compounds on silica gel-G plates with layer thickness of 0.3 mm. All compounds gave satisfactory C, H, N and S elemental analysis.

†Department of Chemistry, Mahatma Gandhi Mahavidyalaya, Warud-444 906, India.
*Hariom Colony, Near Hollywood Colony, Kathora Road, VMV Post, Amravati-444 604, India.

1,3-Bis (N-phenylthioamido) guanidine (3a)

A mixture of guanidine carbonate (0.05 m), phenylisothiocyanate (0.1 m), acetone (25 mL), ethanol (25 mL) was refluxed for 12 h on a water bath in 1 : 2 molar ratio. The precipitated solid was collected by filtration and recrystallized from aqueous ethanol. Yield (82%), m.p. 182°C.

Properties of Compound (3a)

It is a shining fine yellow crystalline solid having m.p. 182°C. From analytical data, m.f. was found to be $C_{15}H_{15}N_5S_2$. IR spectrum (ν_{max} cm^{-1}) of the compound shows ν(N—H) 3387, ν(C—H) (Ar) 3147, ν(C=N) 1666, ν(C—N) 1395, ν(C=S) grouping 1178, ν(C—S) 746 ν(C=NH) grouping 1575. The PMR spectrum of compound showed signals due to Ar-NH protons at δ 7.92–8.0 ppm, Ar-H protons at δ 6.90 ppm, N—H protons at δ 3.25–3.27 ppm and the signal at δ 2.55 ppm is due to moisture in DMSO-d_6 and δ 1.24 ppm is due to DMSO. Found (Calcd.) (%): C = 53.59 (54.71), H = 3.88, (4.56) N = 21.05 (21.27), S = 19.27 (19.45). From these spectral, elemental and chemical data the compound **(3a)** is 1,3-bis(N-phenylthioamido) guanidine.

Similarly others compounds **(3b–3f)** were synthesised by the above mentioned method and listed in Table-1.

TABLE-1
PHYSICAL DATA AND ELEMENTAL ANALYSIS OF THE COMPOUNDS (3b–f)

Compd.	R	m.f.	Yield (%)	m.p. (°C)	Elemental analysis Found (Calcd.) %	
					N	S
3b	*p*-chlorophenyl	$C_{15}H_{13}N_5S_2Cl_2$	82	194	17.59 (17.63)	16.07 (16.12)
3c	*p*-tolyl	$C_{16}H_{16}N_5S_2$	72	189	20.41 (20.47)	18.63 (18.71)
3d	ethyl	$C_7H_{15}N_5S_2$	68	172	30.92 (31.04)	27.39 (27.46)
3e	methyl	$C_5H_{11}N_5S_2$	82	160	34.03 (34.15)	31.13 (31.21)
3f	*t*-butyl	$C_7H_{14}N_5S_2$	62	169	30.12 (30.17)	27.52 (27.59)

ACKNOWLEDGEMENTS

Authors are thankful to Dr. D.M. Ambhore, Principal and J.B. Devhade, Head, Department of Chemistry, Jijamata Mahavidyalaya, Buldhana for providing facilities. Authors are also thankful to Dr. V.N. Ingale, Head, Department of Chemistry, Nagpur University, Nagpur for providing UV spectra. They are also thankful to S.A.I.F., C.I.L. Punjab University, Chandigarh for providing IR, NMR spectra and elemental analysis.

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(*Received*: 27 May 2005; *Accepted*: 2 May 2006) AJC-4860