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Improved Synthesis of Arylguanamines

Reaction of Aromatic Nitriles with Dicyandiamide in Presence of Sodium Ethoxide

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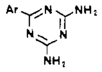
Several new arylguanamines were prepared by interaction of aromatic nitriles with dicyandiamide in the presence of sodium ethoxide.

In connection with a research program involving the synthesis of triazines (*1, 2, 3, 6*), the synthesis of arylguanamines was reinvestigated. The base-catalyzed reaction of dicyandiamide with alkyl or aryl nitriles for the synthesis of 2,4-diamino-6-alkyl-(or aryl)-1,3,5-triazines (guanamines) under drastic conditions is known (*5, 7*). Steric factors influence this reaction and consequently *o*-tolunitrile fails to give the appropriate guanamine (*5*).

Sodium ethoxide was successfully used as a catalyst. Good yields of arylguanamines were obtained (Table I).

All guanamines obtained exhibit characteristic infrared absorption bands at 3 to 3.2 microns for amino groups and bands of strong intensity at 6.1 to 6.3 microns for C=C and C=N.

Table I. Arylguanamine Yields

			NMR Chemical Shift (δ), P.P.M.	
Ar[a]	 M.P., °C.	Yield, %	NH_2 protons	Aromatic protons
C_6H_5	228[b]	27	6.65	7.6–7.75
o-FC_6H_4	247	21	6.50	7.10
m-FC_6H_4	249	66	6.50	7.15–7.8
p-FC_6H_4	249	69	6.55	7.3–8.1
o-ClC_6H_4	232	65	6.50	7.0
m-ClC_6H_4	277	80	6.62	6.4–8.0
p-ClC_6H_4	251[c]	79	6.50	7.3–8.07
o-BrC_6H_4	249	67	6.50	7.25–
m-BrC_6H_4	233	78	6.50	7.25–7.8
p-BrC_6H_4	264	71	6.50	7.2–8.0
o-$CH_3C_6H_4$	202	9	6.50	7.2–8.0
p-$CH_3C_6H_4$	244[d]	27	6.60	7.0–8.0
o-$CF_3C_6H_4$	233	50	6.50	7.4
m-$CF_3C_6H_4$	210	86	6.50	7.4–8.2

[a] C, H, and N analyses for compounds submitted to review and are in accord with theory. [b] (*5*) 226°. [c] (*4*) 226–27°. [d] (*4*) 252–53°. [e] (*6*) 240°.

Other distinguishing bands peculiar to each structure are present. Known compounds had spectra identical with those of authentic samples prepared by other methods (*5, 7*).

The NMR spectrum for each compound is in agreement with that predicted for the protons of the group characteristic of the particular structure. NMR data are tabulated in Table I.

EXPERIMENTAL

Melting points were taken on a Kofler hot-stage microscope and are uncorrected. The infrared spectra were determined with a Leitz Model III spectrograph, using potassium bromide disks. The NMR spectra were determined on a Varian A 60 A instrument at room temperature in dimethylsulfoxide, using TMS as standard.

2,4-Diamino-6-aryl-1,3,5-triazines (Arylguanamines). In all cases 0.01 mole of arylnitrile and 0.02 mole of dicyandiamide in sodium ethoxide solution (prepared by dissolving 0.015 atom of sodium in 35 ml of absolute ethanol were refluxed for 15 hours. The solution obtained was diluted with water and neutralized with acetic acid. The guanamines precipitated were recrystallized from alcohol or dioxane.

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