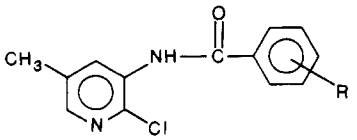
3-(Substituted benzamido)-2-chloro-5-methylpyridines

Frank L. Setliff* and Steve H. Rankin

Department of Chemistry, University of Arkansas at Little Rock, Little Rock, Arkansas 72204

The preparation of 10 3-(substituted benzamido)-2-chloro-5-methylpyridines is described. These compounds were synthesized by the Schotten–Baumann reaction of 3-amino-2-chloro-5-methylpyridine with the appropriately substituted benzoyl chloride. Experimental and spectral data for the 10 compounds are presented.

We recently reported (*1*) the synthesis and characterization of a series of 5-(substituted benzamido)-2-chloro-3-methylpyridines, some of which have been found to possess modest plant fungicidal activity. In this paper we report the preparation of an isomeric series of compounds, namely 3-(substituted benzamido)-2-chloro-5-methylpyridines. These compounds are currently under investigation as possible fungicidal agents.



Experimental Section

Elemental analyses (C, H, N) in agreement with theoretical values were obtained by Galbraith Laboratories, Knoxville, TN, and were submitted for review. Melting points were taken on a Mel-Temp apparatus and are uncorrected. Infrared spectra were obtained on a Perkin-Elmer Model 1430 spectrophotometer equipped with a 7300 data station, with samples prepared as KBr disks (Table I). Proton nuclear magnetic resonance spectra were obtained in deuteriochloroform on a Varian Em-360 instrument with tetramethylsilane as internal standard.

Benzamido Derivative Formation: General Procedure. A mixture of 3-amino-2-chloro-5-methylpyridine (*2*) (0.5 g, 0.0035 mol), the appropriately ring-substituted benzoyl chloride (1.0 mL), and 10% sodium hydroxide (10 mL) in a 25-mL glass-stoppered flask was agitated vigorously on a mechanical shaker for 15 min. In some cases it was necessary to stop the shaker intermittently in order to pulverize the oily solid with a spatula. The resulting solid was filtered, washed liberally with cold water, and recrystallized twice from aqueous ethanol.

Table I. Experimental and Spectral Data[a] for 3-(Substituted benzamido)-2-chloro-5-methylpyridines

compd	R	yield, %	mp, °C	ν (IR), cm^{-1} N–H	ν (IR), cm^{-1} C=O
I	*m*-Br	90	119–121	3269	1655
II	*o*-Cl	61	114–115	3222	1654
III	*p*-Cl	68	147–148	3280	1650
IV	*m*-Cl	76	109–110	3268	1658
V	*p*-F	74	131–132	3290	1653
VI	*o*-F	71	101–102	3413	1685
VII	*o*-Br	74	147–148	3264	1670
VIII	*m*-F	60	112–113	3310	1652
IX	*p*-CF_3	55	145–146	3293	1654
X	*p*-Br	59	149–150	3279	1649

[a] Proton NMR spectra for all compounds revealed a 3 H singlet for the methyl protons in the range δ 2.20–2.40, and the aromatic and amido protons as a composite 7 H multiplet in the range δ 7.00–8.90.

Registry No. **2**, 34552-13-1; **I**, 112841-05-1; **II**, 112841-06-2; **III**, 112841-07-3; **IV**, 112841-08-4; **V**, 112841-09-5; **VI**, 112841-10-8; **VII**, 112841-11-9; **VIII**, 112841-12-0; **IX**, 112841-13-1; **X**, 112841-14-2; *m*-BrC_6H_4COCl, 1711-09-7; *o*-ClC_6H_4COCl, 609-65-4; *p*-ClC_6H_4COCl, 122-01-0; *m*-ClC_6H_4COCl, 618-46-2; *p*-FC_6H_4COCl, 403-43-0; *o*-FC_6H_4COCl, 393-52-2; *o*-BrC_6H_4COCl, 7154-66-7; *m*-FC_6H_4COCl, 1711-07-5; *p*-$CF_3C_6H_4COCl$, 329-15-7; *p*-BrC_6H_4COCl, 586-75-4.

Literature Cited

(1) Setliff, F.; Palmer, H. *J. Chem. Eng. Data* **1987**, *32*, 393.
(2) Setliff, F. *Org. Prep. Proc. Int.* **1971**, *3*, 217.

Received for review August 10, 1987. Accepted November 30, 1987.

0021-9568/88/1733-0223$01.50/0