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

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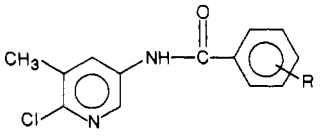
The preparation of 10 5-(substituted benzamido)-2-chloro-3-methylpyridines is described. These compounds were synthesized by the Schotten-Baumann reaction of 5-amino-2-chloro-3-methylpyridine with the appropriately substituted benzoyl chloride. Experimental and spectral data for the 10 compounds are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

5-Benzamido-2-chloro-3methylpyridine (**1**) has recently been shown to possess low level herbicidal properties toward certain types of barnyard grasses. In an effort to induce more significant activity, we have prepared a series of derivatives with a variety of substituents on the benzene ring.



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 (Table I). Infrared spectra were obtained on a Perkin-Elmer 337 spectrophotometer with samples prepared as KBr disks. Proton nuclear magnetic resonance spectra were obtained in either deuteriochloroform or acetone-d_6 on a Varian Em-360 instrument with tetramethylsilane as internal standard.

Benzamido Derivative Formation: General Procedure. A mixture of 5-amino-2-chloro-3-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.

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

compd	R	yield, %	mp, °C	IR, ν, cm^{-1}
I	*m*-Br	61	146-147	3300, 1658, 1582, 1527, 1460, 1299, 1205, 1147, 1053, 877.2, 799.4, 720.5
II	*o*-Cl	86	133-134	3236, 1658, 1587, 1506, 1385, 1302, 1044, 867.3, 736.4, 718.9
III	*p*-Cl	87	193-194	3300, 1675, 3268, 1648, 1592, 1524, 1486, 1272, 1053, 990.1, 873.4 839.6, 741.8, 716.9
IV	*m*-Cl	86	144-146	3268, 1648, 1527, 1412, 1311, 1269, 1054, 881.1, 805.2, 778.2, 711.7
V	*p*-F	97	193-194	3290, 1653, 1603, 1582, 1531, 1506, 1235, 1224, 1136, 1050, 873.4, 847.5, 719.4
VI	*o*-F	88	116-117	3378, 1664, 1580, 1520, 1481, 1449, 1294, 1224, 1149, 1071, 1048, 892.9, 871, 809.7, 765.5, 740.7, 714.8
VII	*m*-NO_2	78	224-226	3290, 1661, 1531, 1462, 1403, 1350, 1319, 1284, 1148, 1052, 897.7, 848.9, 807, 717.9
VIII	*m*-F	86	144-145	3322, 1650, 1592, 1522, 1290, 1182, 1058, 829.9, 827.8, 793.7, 718.9
IX	*p*-CF_3	87	207-209	3311, 1658, 1605, 1585, 1534, 1464, 1414, 1332, 1138, 1100, 1080, 1000, 874.9, 849.6, 832.6, 764.5, 722
X	*p*-Br	75	188-190	3290, 1648, 1592, 1524, 1401, 1309, 1151, 1053, 996, 873.4, 829.2, 740.7, 720.5

[a] Proton NMR spectra for all compounds revealed a 3 H singlet for the methyl protons in the range δ 2.30 to 2.50 and the aromatic and amido protons as a composite 7 H multiplet in the range δ 7.00 to 8.90.

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The resulting solid was filtered, washed liberally with cold water, and recrystallized twice from aqueous ethanol.

Registry No. 2, 38186-82-2; I, 108666-31-5; II, 108666-32-6; III, 108666-33-7; IV, 108666-34-8; V, 108666-35-9; VI, 108666-36-0; VII, 108666-37-1; IX, 108666-38-2; X, 108666-39-3; oXI, 108666-40-6; 3-BrC_6H_4COCl, 1711-09-7; 2-ClC_6H_4COCl, 609-65-4; 4-ClC_6H_4COCl, 122-01-0; 3-ClC_6H_4COCl, 618-46-2; 4-FC_6H_4FCOCl, 403-43-0; 2-FC_6H_4COCl, 393-52-2; 3-$O_2NC_6H_4COCl$, 121-90-4; 3-FC_6H_4COCl, 1711-07-5; 4-$F_3CC_6H_4COCl$, 329-15-7; 4-BrC_6H_4COCl, 586-75-4.

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