

化学试剂，2007, 29(5), 303-304

\[ N - \square - 3 - \square - 6 (3, 5 - \square - \square - 1 - \square) \]
\[ \square - 2 - \square - \square - 3 - \square - 6 (3, 5 - \square - \square - 1 - \square) \]

冯宇1, 尹显洪1, 杨宁1, 莫艳梅1, 赵凯2, 胡辉2
(1. \( N \square - 3 \square - 6 (3, 5 \square - \square - 1 \square) \) \( 530006 \)～
2. \( N \square - 3 \square - 6 (3, 5 \square - \square - 1 \square) \) \( 530006 \))

\[ 0621.3 \quad \square \square : A \quad 0258-3283(2007)05-0303-02 \]

1. 2 \( N - \square - 3 - \square - 6 (3, 5 - \square - \square - 1 - \square) \)

\[ 10 \text{g (0.052 mol)} \quad 3. \quad 6 - \square - \square - 2 - \square - \square - (2) \]

\[ 250 \text{mL} \quad 50 \text{mL} \quad 6 h \quad 30 \text{mL} \quad 3 \text{g (0.052 mol)} \quad 50 \text{mL} \]

\[ 2 \text{h} \quad 12.5 \text{g} \quad 90\% \quad 130-132\degree \text{C} \quad 3 \text{mL} \quad 300 \text{N} \quad 300 \text{C} \quad 1 \text{H} 690 \text{C} \quad 1 \text{H} 520 \text{C} \quad 1 \text{H} 500 \text{C} \quad 1 \text{H} 490 \text{C} \]

1. 3 \( N - \square - 3 - \square - 6 (3, 5 - \square - \square - 1 - \square) \)

\[ \square - \square : \quad \text{100 mL} \quad 85\% \quad \text{2:1} \quad 1.5 - 2 \text{h} \]

1. 鲁尔默 550 (800 W) ；XT-4 (800 W) ；Varian
2. Mercury-300 (TMS) ；CDCl3 (800 W) ；Biflex 3 (800 W) ；
1.4 3,5-dimethyl-4-ethylphenol (0.0075 mol) was dissolved in 30 mL of 5% NaOH (0.01 mol) and 3 mL of 10% DMF was added. The mixture was heated under reflux for 30 min. The precipitated product was filtered, washed with water, and dried in vacuum (V : V = 3 : 1). The yield was 60% (92 g). IR (KBr), ν, cm⁻¹: 3 300 (N–H); 3 100 (C–H); 2 820 (C–H); 1 650 (C=O); 1 520 (C=O); 1 500 (C=C). ¹H NMR (CDCl₃, δ: 8.05 (d, 1H, CH of pyridine)), 7.9 (d, 1H, CH of pyridine), 7.71 (d, 2H, H of pyridine), 7.39 (m, 2H, H of pyridine), 7.15 (t, 1H, CH of pyridine), 2.3 (s, 3H, –CH₃ of pyrazole), 2.76 (s, 3H, –CH₃ of pyrazole), 6.08 (s, 1H, CH of pyrazole), 9.54 (s, 1H, –NH–). ¹³C NMR (CDCl₃, 300 MHz), δ: 161.0 (C=O), 151.04, 149.83, 140.89, 128.47, 119.01 (5), 137.59, 129.19 (2C), 124.62, 119.49 (2C), 143.17, 142.82, 110.5 (1), 13.56 (–CH₃), 15.53 (–CH₂), 327 (M⁺ + 1). MS, m/z: 327 (M⁺ + 1), 110 (5), 10 (5), 87 (100), 77 (100), 65 (100), 53 (100), 41 (100), 39 (100), 31 (100), 29 (100), 27 (100), 19 (100), 17 (100), 15 (100), 13 (100), 11 (100), 9 (100), 7 (100), 5 (100), 3 (100), 2 (100), 1 (100).

Synthesis and characterization of 3-chloro-6-(3, 5-dimethyl-1H-pyrazol-1-yl)-N-phenylpicolinamide and 3-chloro-6-(3, 5-dimethyl-1H-pyrazol-1-yl) picolinic acid. FENG Yu¹, YING Xian-hong², YANG Ning³, MO Yan-mei², ZHAO Kai², ZHU Jie² (1. College of Chemistry and Ecological Engineering, Guangxi University for Nationalities, Nanning 530006). 2. College of Chemistry and Chemical Engineering, Guangxi University, Nanning 530006. China; Huanmei Shi. 2007, 29(5), 303–304.

Abstract: Through the four-step reactions including amidization diazoyl substitution cyclocondensation and hydrolysis, two new organic heterocyclic ligands, i.e., 3-chloro-6-(3, 5-dimethyl-1H-pyrazol-1-yl)-N-phenylpicolinamide and 3-chloro-6-(3, 5-dimethyl-1H-pyrazol-1-yl) picolinic acid were synthesized using 3, 6-dichloro-pyridine-2-methane acid as the starting materials and their chemical structures were characterized by IR, ¹H NMR, ¹³C NMR and MS.

Key words: The derivatives of pyridine synthesis hydrolysis...