

3-tert-butoxycarbonylamino-pyridine-2-carboxylic Acid

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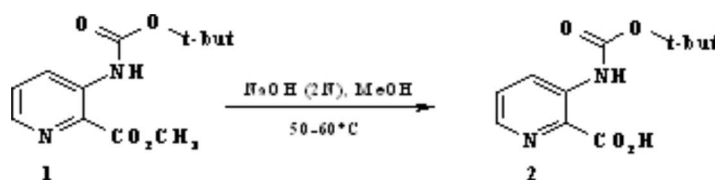
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To the carbamic acid tert-butylester 1 (1.03 g, 4.08 mmol) in methanol (15 ml) was added 1.4 equivalents of NaOH (2N). The mixture was stirred at 50-60°C for 6 hours. After cooling to room temperature, the solvent was removed under reduce pressure. The residue was dissolved in water and acidified with HCl (15%) to pH 2 and extracted with dichloromethane (3 x 15 ml). The organic layer fractions were combined, dried (MgSO₄) and the solvent was evaporated in vacuum to give a white solid 2 (0.924 g, 95%).

Mp 154°C (White powder from Ethyl acetate/petroleum ether)

IR (KBr): 2994, 1727, 1677 cm⁻¹

¹H-NMR (300 MHz, DMSO-d₆, δ, ppm): 1.48 (s, 9H, CH₃), 7.69 (dd, 1H, *J* = 8.5, 4.5 Hz, H-5), 8.31 (dd, 1H, *J* = 4.5, 1.3 Hz, H-4), 8.75 (dd, 1H, *J* = 8.5, 1.3 Hz, H-6), 10.79 (s, 1H, NH).

¹³C- NMR (75 MHz, DMSO-d₆, δ, ppm): 27.82 (3CH₃), 80.85 (C(CH₃)₃), 127.80, 128.31, 132.13, 138.75, 140.25, 152.00, 167.04.

MS m/z: 239 [M+1], 183, 165, 147, 139, 121

Elemental analysis: Calculated C₁₁H₁₄N₂O₄: C, 55.46; H, 5.92; N, 11.76 Found: C, 55.36; H, 5.86; N, 11.79.

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