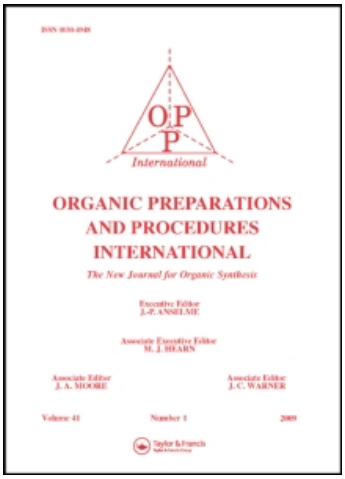

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AN IMPROVED SYNTHESIS OF 2-IODO-5-NITROPYRIDINE

Frank L. Setliff[a]
[a] Department of Chemistry, University of Arkansas at Little Rock, Little Rock, Arkansas

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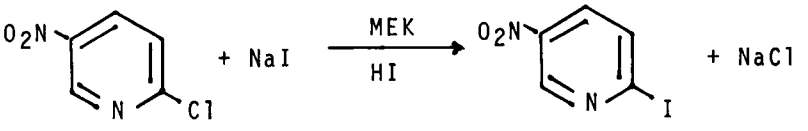
AN IMPROVED SYNTHESIS OF 2-IODO-5-NITROPYRIDINE

Frank L. Setliff

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

Previous communications report the preparation of 2-iodo-5-nitropyridine in 14 and 30% yields,[1,2] by diazotization of 2-amino-5-nitropyridine in the presence of potassium iodide or in the presence of hydriodic acid respectively. We have found that 2-chloro-5-nitropyridine (commercially available and inexpensive) undergoes smooth chloride-iodide exchange with sodium iodide and hydriodic acid in refluxing methyl ethyl ketone to produce the title compound in 60-70% yields. In the absence of hydriodic acid starting material is quantitatively recovered. This evidence suggests that further activation of the substrate by protonation of the ring nitrogen is apparently necessary.



EXPERIMENTAL

A mixture of 2-chloro-5-nitropyridine (4.0 g, 0.0252 mole), sodium iodide (11.0 g, 0.22 mole), 57% hydriodic acid (2 ml), water (3 ml), and methyl ethyl ketone (55 ml) was stirred under gentle reflux for 16 hr. The reaction mixture was cooled, filtered free of sodium chloride, and the dark filtrate was evaporated to dryness using a rotary evaporator. The residue was suspended in water (75 ml) and made basic with 10% sodium hydroxide. Sodium bisulfite (5.0 g) was then added and the suspension was stirred overnight. The yellow-green crude product was filtered and recrystallized from acetone-water to yield 4.25 g (68%) of 2-iodo-5-nitropyridine, mp. 164-165°, lit.[1] mp. 165-166°. IR(KBr): 3080 (w), 3030 (w), 1580 (s), 1550 (s), 1500 (s), 1470 (w), 1430 (s), 1325 (s), 1260 (m), 1235 (m), 1050 (s), 1000 (m), 925 (w), 840 (s), 735 (m), 617 (w) cm^{-1}. Nmr (acetone d_6): δ 9.33, 1 H, m, H_6; δ 8.13, 2 H, m, $H_3 + H_4$.

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