

This article was downloaded by:

On: 27 January 2011

Access details: *Access Details: Free Access*

Publisher *Taylor & Francis*

Informa Ltd Registered in England and Wales Registered Number: 1072954 Registered office: Mortimer House, 37-41 Mortimer Street, London W1T 3JH, UK



## Organic Preparations and Procedures International

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t902189982>

### AN IMPROVED SYNTHESIS OF 2-IODO-5-NITROPYRIDINE

Frank L. Setliff<sup>a</sup>

<sup>a</sup> Department of Chemistry, University of Arkansas at Little Rock, Little Rock, Arkansas

**To cite this Article** Setliff, Frank L.(1973) 'AN IMPROVED SYNTHESIS OF 2-IODO-5-NITROPYRIDINE', *Organic Preparations and Procedures International*, 5: 6, 305 – 306

**To link to this Article:** DOI: 10.1080/00304947309356862

**URL:** <http://dx.doi.org/10.1080/00304947309356862>

PLEASE SCROLL DOWN FOR ARTICLE

Full terms and conditions of use: <http://www.informaworld.com/terms-and-conditions-of-access.pdf>

This article may be used for research, teaching and private study purposes. Any substantial or systematic reproduction, re-distribution, re-selling, loan or sub-licensing, systematic supply or distribution in any form to anyone is expressly forbidden.

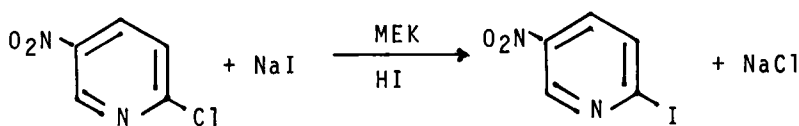
The publisher does not give any warranty express or implied or make any representation that the contents will be complete or accurate or up to date. The accuracy of any instructions, formulae and drug doses should be independently verified with primary sources. The publisher shall not be liable for any loss, actions, claims, proceedings, demand or costs or damages whatsoever or howsoever caused arising directly or indirectly in connection with or arising out of the use of this material.

## 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,<sup>1,2</sup> 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<sup>o</sup>, lit.<sup>1</sup> mp. 165-166<sup>o</sup>. 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<sup>-1</sup>. Nmr (acetone d<sub>6</sub>):  $\delta$  9.33, 1 H, m, H<sub>6</sub>;  $\delta$  8.13, 2 H, m, H<sub>3</sub> + H<sub>4</sub>.

## REFERENCES

1. F. Case, J. Am. Chem. Soc., 68, 2574 (1946).
2. W.T. Caldwell, F.T. Tyson, and L. Lauer, *ibid.*, 66, 1479 (1944).

(Received August 14, 1973)