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### 2-FLUORO-5-NITRO-3-PICOLINE AND 6-FLUORO-5-NITRO-3-PICOLINE BY CHLORIDE-FLUORIDE EXCHANGE

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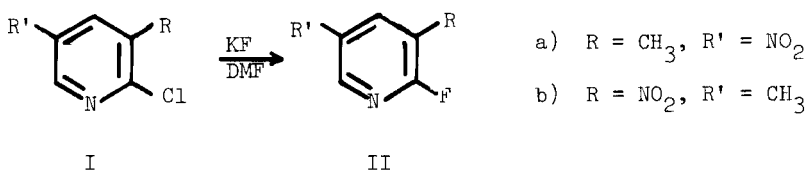
## OPPI BRIEFS

### 2-FLUORO-5-NITRO-3-PICOLINE AND 6-FLUORO-5-NITRO-3-PICOLINE BY CHLORIDE-FLUORIDE EXCHANGE

Submitted by F. L. Setliff\* and D. J. DeFoggi  
(12/19/77)

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The preparation of 2-fluoronitropyridines by diazotization of 2-amino nitropyridines characteristically gives very low yields.<sup>1</sup> 2-Chloro-5-nitro-3-picoline (Ia)<sup>2</sup> and 6-chloro-5-nitro-3-picoline (Ib)<sup>3</sup> have been successfully converted to the previously unreported title compounds IIa and IIb in 60-70% yields by nucleophilic displacement of chloride with fluoride.<sup>4</sup>



#### EXPERIMENTAL

All melting points are uncorrected. Infrared spectra were obtained in KBr using a Perkin-Elmer 337 spectrophotometer. Proton nuclear magnetic resonance spectra were obtained on a Jeolco C-60 HL instrument in deuteriochloroform with TMS as internal standard. Elemental analyses were performed by Heterocyclic Chemical Corporation, Harrisonville, MO.

2-Fluoro-5-nitro-3-picoline (IIa).- A mixture of 2-chloro-5-nitro-3-picoline (5.0 g, 0.029 mole), anhydrous potassium fluoride (5.1 g, 0.88 mole)

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and anhydrous dimethylformamide (5.0 ml) was heated under reflux for 6 hrs. The hot reaction mixture was poured over crushed ice, saturated with ammonium chloride, and subjected to indirect steam distillation. The crude product separated as a yellow solid on overnight refrigeration of the steam distillate. Recrystallization from methylcyclohexane afforded pure IIa as small off-white needles, 3.1 g (68.5%), mp. 41-42°. IR ( $\nu_{\max}$ ): 1618, 1602, 1527, 1464, 1449, 1351, 1234, 1149, 1087, 936, 922, 809, 752, 639  $\text{cm}^{-1}$ . NMR:  $\delta$  2.53 (singlet, 3H, methyl protons); 8.46 (doublet of multiplets, 1H, ring proton at C<sub>4</sub>); 8.96 (broad singlet, 1H, ring proton adjacent to ring nitrogen).

Anal. Calcd for C<sub>6</sub>H<sub>5</sub>N<sub>2</sub>O<sub>2</sub>F: C, 46.15; H, 3.21; N, 17.95.

Found: C, 46.20; H, 3.30; N, 17.99.

6-Fluoro-5-nitro-3-picoline (IIb), mp. 39-40°, was similarly obtained in 63% yield. IR ( $\nu_{\max}$ ): 1607, 1575, 1534, 1466, 1385, 1351, 1255, 1227, 1169, 1075, 930, 893, 812, 763, 750, 662  $\text{cm}^{-1}$ .

NMR:  $\delta$  2.58 (singlet, 3H, methyl protons); 8.38 (multiplet, 2H, ring protons).

Anal. Calcd. for C<sub>6</sub>H<sub>5</sub>N<sub>2</sub>O<sub>2</sub>F: C, 46.15; H, 3.21; N, 17.95.

Found: C, 46.35; H, 3.43; N, 18.22.

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