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## Organic Preparations and Procedures International

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### 1-BROMOPHENANTHRIDINE

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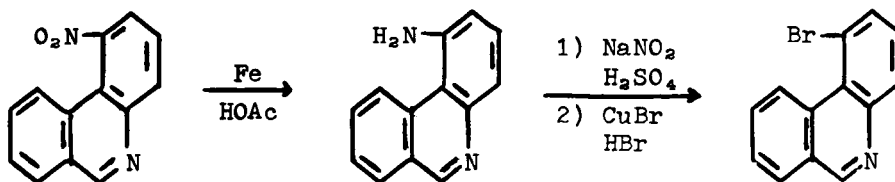
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1-BROMOPHENANTHRIDINE

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1-Bromophenanthridine has been synthesized<sup>1</sup> from 1-carboxyphenanthridone in a number of steps. We wish to report an alternative synthesis.

1-Nitrophenanthridine, which is one of the isomers obtained upon nitration of phenanthridine,<sup>2</sup> was reduced to 1-aminophenanthridine. The 1-aminophenanthridine was converted to 1-bromophenanthridine through its diazonium salt.



EXPERIMENTAL

1-Aminophenanthridine. - A mixture of 0.9 g. (0.0040 mole) of 1-nitrophenanthridine<sup>2</sup> (from chromatography of the mixture obtained upon nitration of phenanthridine), 1.8 g. of iron powder, 45 ml. of water, and 0.9 ml. of 2 N acetic acid was heated for 4 hr. on a steam bath. Extraction with boiling benzene gave 0.75 g. (97%) of amine, mp. 115-116.5°, lit.<sup>2</sup> mp. 115.5-117°.

1-Bromophenanthridine. - To a mixture of 1.2 g. of sulfuric acid and 0.75 g. (0.0038 mole) of 1-aminophenanthridine at 0° was slowly added 0.35 g. of sodium nitrite in a minimum of water. This mixture was then added to a solution of 0.58 g. of cuprous bromide in 1.62 g.

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of 40% hydrobromic acid at 0° and stirred for 4 hrs. The mixture was gradually brought to 60°, diluted with water, and filtered. The solid was washed with 5% sodium hydroxide and water and recrystallized from ethanol to give 0.6 g. (62%) of 1-bromophenanthridine, mp. 101-102°, lit.<sup>1</sup> mp. 97-99°. The spectral data were consistent with those previously reported.<sup>1</sup>

Anal. Calcd. for C<sub>13</sub>H<sub>8</sub>BrN: C, 60.49; H, 3.12; N, 5.43. Found: C, 60.22; H, 3.07; N, 5.64.

#### REFERENCES

1. G.S. Chandler, J.L. Huppatz, R. A. Jones, and W.H.F. Sasse, Aust. J. Chem. 20, 2037 (1967).
2. A.C. Caldwell and L.P. Walls, J. Chem. Soc., 2156 (1952).

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