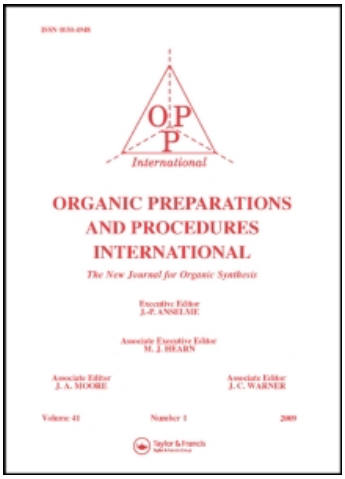

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A FACILE SYNTHESIS OF 2, 2'-BIPYRIDYL-3, 3'-DICARBOXYLIC ACID

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A FACILE SYNTHESIS OF 2,2'-BIPYRIDYL-3,3'-DICARBOXYLIC ACID

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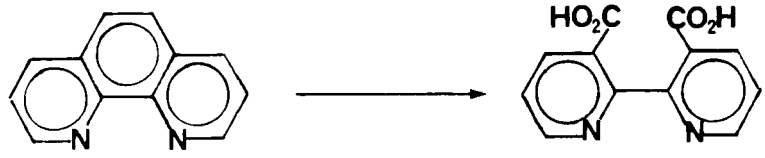
The synthesis of 2,2'-bipyridyl-3,3'-dicarboxylic acid (binicotinic acid) by the oxidation of 1,10-phenanthroline with alkaline permanganate was first described by Smith and Inglett,[1] who reported yields of 80%. The preparation of binicotinic acid by this method has subsequently been reported by several workers[2] with yields varying from 50-85%. We have been unable to reproduce the high yield reported by Smith and Inglett. The main loss in yield occurs in the conversion of the silver salt of binicotinic acid to the free acid by treatment with hydrogen sulphide.



We describe here a simplified preparation of binicotinic acid in good yield (75-80%) with the elimination of the tedious work-up procedure.

EXPERIMENTAL

1,10-Phenanthroline monohydrate (16.0 g, 0.081 mol), sodium hydroxide (6.4 g, 0.16 mol) and potassium permanganate (38.0 g, 0.24 mol) were dissolved in water (700 ml) and the mixture was boiled in a 1.5 l. Erlenmeyer flask with stirring

for 2.5 hrs. The precipitate of manganese dioxide was removed by filtration and the filtrate concentrated to ca. 300 ml. The pH of the solution was adjusted to ca. 2 with conc. hydrochloric acid and decolourising charcoal was added. The mixture was boiled, filtered and concentrated to ca. 200 ml. The white needles of binicotinic acid (75-80%) were filtered, washed with cold water and then cold ethanol and dried in vacuo over P_2O_5, mp. 262° (dec.), lit.[1] 260-261° (dec.).

Anal. Calcd for $C_{12}H_8N_2O_4$: C, 59.0; H, 3.3

Found: C, 58.9; H, 3.3.

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