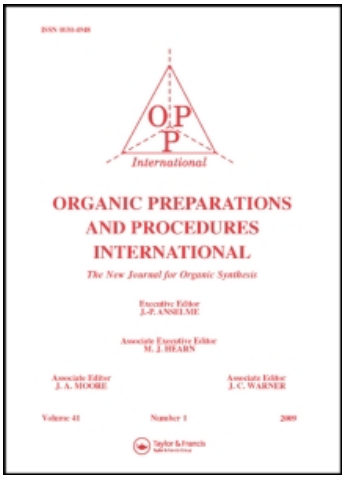

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AN IMPROVED SYNTHESIS OF 2,2'-DIPYRIDYL-1-OXIDE

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as stirring was continued for an additional 3 hrs. The suspension was allowed to stand overnight and filtered to remove the pyridine hydrochloride, which was washed with methylene chloride. The combined filtrates were washed with H_2O and a total of 3 ℓ. of 5% HCl and dried over $MgSO_4$. The solvent was removed on a rotary evaporator at 35-40° and the residual liquid (2.258 kg) was distilled through a 12" Vigreux column. After removal of a small forerun, the main fraction, 2.115 kg (93.5% yield), was collected as a colorless liquid, bp. 71-74°/0.2 mm, n_D^{24} 1.481. The material was homogeneous to GC and no additional peaks were observed in its NMR spectrum.

AN IMPROVED SYNTHESIS OF 2,2'-DIPYRIDYL-1-OXIDE

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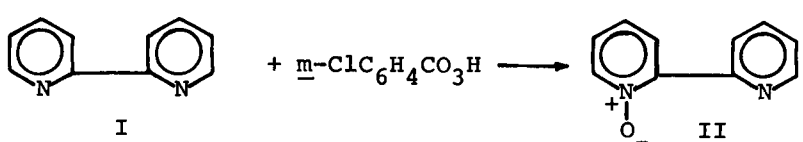
2,2'-Dipyridyl-1-oxide (II),[1,2] previously isolated as a brown hygroscopic solid,[2,3] from the oxidation of 2,2'-dipyridyl (I) with 30% hydrogen peroxide in acetic acid, can be obtained in 50-55% yield from the oxidation of I with m-chloroperbenzoic acid[4] in chloroform.



PROCEDURE

To a stirred ice-cold chloroform (15 ml.) solution of 2,2'-dipyridyl (1.99 g., 12.8 mmol) was added over a period of 2 hrs. a solution of 2.98 g. (1.72 mmol) of m-chloroperbenzoic acid (85% peracid) in 60 ml. of

chloroform. The solution was then stirred for 1 hr. at room temperature and 10 g. of basic alumina (chromatographic grade) was added and the mixture evaporated to dryness (rotary evaporator). The powder was then added to the top of a 12" x 0.5" alumina column; elution with 3:1 benzene-chloroform (v/v) gave II as an oil. Addition of ether followed by slow concentration gave 1.10-1.22 g. (50-55%) of pure II, mp. 59-61°, lit.[4] 58.5-59.5°.

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^{13}C-LABELLED ALIPHATIC NITRILES AND KETONES

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^{13}C-Labelled aliphatic nitriles and ketones have been prepared as shown.

$$R\text{-}X + K^{13}CN \longrightarrow R^{13}CN \xrightarrow{MeMgI} \xrightarrow{H_2O,\ H^+} R^{13}COCH_3$$