CARBONYLATION OF 2-AMINOPYRIDINE WITH CARBON MONOXIDE

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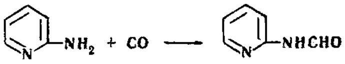
α-Aminopyridine is smoothly carbonylated by carbon monoxide under pressure to give α-formamidopyridine. Phenothiazine and acridone are not carbonylated under these conditions.

Continuing our studies of the carbonylation of nitrogen heterocycles [1,2], in this paper we have investigated the behavior of α-aminopyridine, phenothiazine, and acridine with respect to carbonylation with carbon monoxide using sodium methoxide in methanol as the catalyst. α-Aminopyridine reacts with carbon monoxide at 60°C and 125 atm, and α-formamidopyridine is formed in 64% yield after heating for 0.5 h.



Virtually no resin formation is observed at 60°. An increase in the temperature promotes an increase in the conversion of the starting aminopyridine but reduces the yield of the formamide because of side processes leading to resinification. A change in pressure above 125 atm and an increase in the heating time beyond 0.5 h have similar effects.

Phenothiazine is not carbonylated by carbon monoxide in the presence of sodium methoxide at 40-120° and 140-250 atm. It also could not be formylated with formic acid or methyl formate. Acridone is not formylated under similar conditions.

EXPERIMENTAL

A 0.25-liter rotating autoclave was charged with 1.88 g (0.02 g-mole) of α-aminopyridine and 25 ml of absolute methanol containing 0.46 g of sodium metal, CO was added to a pressure of 125 atm, and the mixture was heated at 60° for 30 min. The autoclave was cooled, the solvent was removed, and the solid (2.5 g) was washed with benzene and dissolved in ether. These two solutions yielded 1.4 g (64%) of α-formamidopyridine with mp 76-77°. The picrate melted at 208°. This product did not depress the melting point of α-formamidopyridine obtained by the formylation of α-aminopyridine with formic acid. The structure was also confirmed by elementary analysis and IR spectroscopy.

LITERATURE CITED

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