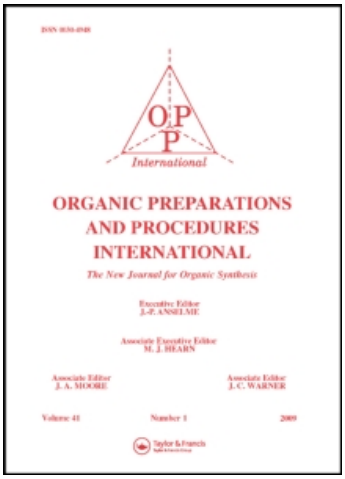

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NOVEL METHODS OF PREPARATION OF 2-OXAZOLIDONES

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NOVEL METHODS OF PREPARATION OF 2-OXAZOLIDONES

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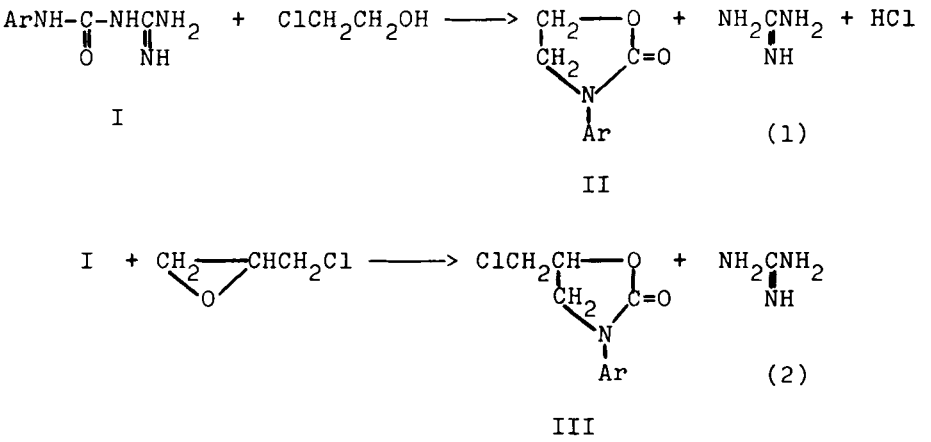

Numerous methods are known for preparation of 2-oxazolidones.[1-15] We are reporting now two new methods of obtaining this class of compounds. Method (1) consists of the reaction of ethylene chlorohydrin and amidine urea derivatives.[16] 3,5-Disubstituted 2-oxazolidones were obtained by method (2). Here epichlorohydrin was used instead of ethylene chlorohydrin. The reactions are carried out in one stage, under atmospheric pressure, without solvent and catalyst, and the yields are good.

EXPERIMENTAL

3-(p-Nitrophenyl)-2-oxazolidone(II,Ar=p-nitrophenyl).

N'-(p-nitrophenyl)-N"-amidine urea (I,Ar=p-nitrophenyl) (7.9 g, 0.035 mole) and ethylene chlorohydrin (75 ml) were refluxed to the complete solution (∿ 4 hrs.). After cooling water (290 ml) was added dropwise. The pale yellow precipitate was washed with water to give 5.3 g (70.2%). One crystallization from ethanol gave an analytical sample, mp. 156-157°.

Anal. Calcd, for $C_9H_8N_2O_4$: C, 51.95; H, 3.87; N, 13.46.
Found: C, 51.68; H, 3.73; N, 13.22.

IR spectrum: the C=O band characteristic for 2-oxazolidones at 1760 cm^{-1}.

NMR spectrum: τ 1.7, 1.9, 2.3, 2.5, and multiplet centered at τ 5.45 protons of the aromatic and oxazolidone ring respectively. (Up to now, II had been prepared by a different route.)

3-(p-Nitrophenyl)-5-chloromethyl-2-oxazolidone(III,Ar=p-nitrophenyl).

II (Ar=p-nitrophenyl) [4.4 g, 0.0197 mole] and epichlorohydrin (30 ml) were refluxed to achieve complete solution (∿ 2 hrs.). After cooling, ethanol (30 ml) was added. The precipitate was washed with ethanol to yield 3.1 g (64%). Crystallization from ethanol or dioxane gave yellow plates, mp. 155-157°.

Anal.

Calcd. for $C_{10}H_9ClN_2O_4$: C, 46.80; H, 3.53; Cl, 13.81; N, 10.91

Found: C, 46.72; H, 3.59; Cl, 13.68; N, 10.95

IR spectrum: C=O band characteristic for 2-oxazolidone ring at 1760 cm^{-1}. The position of chloromethyl group was established by NMR: τ 1.72, 1.9, 2.3, 2.5, protons of the aromatic ring. Multiplet centered at τ 4.9 is due to the single proton adjacent to oxygen in 2-oxazolidone ring.

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