THE CONVERSION OF 3-PHENYLOXAZOLIDINE INTO p, p'-DI(3-OXAZOLIDYL)DIPHENYLMETHANE

S. R. Tulyaganov and F. Yu. Shakirova

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The opening of the oxazolidine ring under the action of organic acid derivatives has been reported in the literature [1].

In a study of the reaction of acetic anhydride with 3-phenyloxazolidine we isolated a colorless crystalline substance which proved, against expectation, to be p, p'-di(3-oxazolidyl)diphenylmethane (I). The I formed was identical with the p, p'-di(3-oxazolidinyl)diphenylmethane obtained from monoethanolaniline and formaldehyde in the presence of hydrochloric acid. When 3-phenyloxazolidine was heated in the absence of acetic anhydride, compound I was again formed. The formation of I can apparently be represented by the following scheme.

$$\begin{array}{cccc} C_{6}H_{5}-N & \stackrel{t^{\circ}}{\longrightarrow} & C_{6}H_{5}-N & + CH_{2}O \\ 2 C_{6}H_{5}-N & \stackrel{t^{\circ}}{\longrightarrow} & C_{6}H_{2}-N & \stackrel{t^{\circ}}{\longrightarrow} & -CH_{2}-CH_{2}-CH_{2}-N & \stackrel{t^{\circ}}{\longrightarrow} & + H_{2}O \end{array}$$

It was impossible to isolate the N-phenylethyleneimine formed as a by-product from the reaction mixture because of its resinification, apparently owing to its sensitivity to the action of heat, air, and various reagents [3].

It is known [4, 5] that p, p'-di(3-oxazolidinyl)diphenylmethane is the basis of phenol-oxazolidine moulding materials. The transformation of 3-phenyloxazolidine into p, p'-di(3-oxazolidinyl)diphenylmethane that we have observed makes it possible when necessary to obtain these materials directly from 3-phenyloxazolidine. Moreover, the formaldehyde liberated during the reaction may accelerate the process. **p. p'-Di(3-oxazolidinyl)diphenylmethane.** A mixture of 10 g (0.07 mole) of 3-phenyloxazolidine and 10 ml (0,1 mole) of acetic anhydride was heated at $120^{\circ}-130^{\circ}$ C for 2 hr. After cooling, the mixture set to a light brown viscous mass. The crystals that deposited were separated off and recrystallized from ethanol. Yield 6.9 g (33%), mp 142° C. Found, %: N 9.10, 9.04. Calculated for $C_{19}H_{22}N_2O_2$, %: N 9.03.

A mixture with an authentic sample obtained by Petrov and Gosteva's method [3] melted without depression. The IR spectra of the two samples were identical.

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Institute of the Chemistry of Plant Substances, AS Uzbek SSR, Tashkent

A NEW METHOD OF OBTAINING 4-QUINAZOLONES

L. I. Samarai, V. A. Bondar, and G. I. Derkach

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The action of phosgene on N-arylamidines in toluene gives 4quinazolones in quantitative yields. The reaction probably takes place with the formation of isocyanates of carbimidic acids which are immediately converted into 4-quinazolones (cf. [1, 2])



For example, a solution or a suspension of 0.2 mole of the amine in toluene is added slowly with stirring to a solution 0.04 mole of phosgene in 20 ml of anhydrous toluene cooled to -15° C. With the continuous passage of phosgene, the reaction mixture is gradually heated to $130^{\circ}-140^{\circ}$ C (bath temperature) and is kept there for 1 hr. The solvent is distilled off in vacuum to half the original volume and the 4-quinazolone that has precipitated is separated off, washed with ether, and dried. Yield 90-93%.

2-Trichloromethyl-4-quinazolone (Ia), mp 211° - 212° C; the compound gave no depression of the melting point in admixture with a sample that we had obtained previously [1].

6-Methoxy-2-trichloromethyl-4-quinazolone (Ib), mp $206^{\circ}-207^{\circ}$ C (from a mixture of methanol and water). Found, \Re : C 41.29; H 2.43. Calculated for $C_{10}H_7Cl_3N_2O_2$, \Re : C 40.91; H 2.40.