REACTIONS THAT OCCUR DURING THE SYNTHESIS OF 3-PHENYL-5-PHENOXYMETHYL-2-N-PHENYLIMINOOXAZOLIDINE

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Previously it was shown [1] that 3-phenyl-5-phenoxymethyl-2-N-phenyliminooxazolidine (I) is formed when diphenylcarbodiimide (DPC) is reacted with an equimolar amount of phenyl glycidyl ether (PGE) in o-dichlorobenzene as the medium.

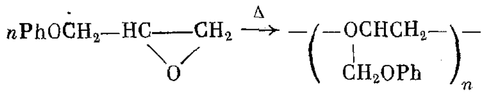
When this reaction is run in the absence of a solvent, the IR spectra of the reaction products have, besides the 1680 cm^{-1} band, which is characteristic for the C=N group of the iminooxazolidine ring, also bands at 1690 cm^{-1}, which can be assigned to the cyclodimerization product of DPC [2], and 1755 cm^{-1}, which is characteristic for the C=O group of the oxazolidone ring [1, 3].

As a result, the reaction of DPC with PGE in the absence of a solvent is nonselective. The formation of the oxazolidone ring is possible if (I) is partially hydrolyzed to 3-phenyl-5-phenoxymethyl-2-oxazolidone (II) in the presence of traces of moisture [1, 4].

However, running the reaction of DPC with equimolar amounts of PGE and water failed to shift the equilibrium toward the formation of (II) (the ratio of the 1680 and 1755 cm^{-1} peaks is the same in both experiments).

The reaction of DPC with a double mole excess of PGE under the same conditions led to a sharp increase in the intensity of the 1755 cm^{-1} band and a decrease in the intensity of the 1680 cm^{-1} band, while with a triple excess of PGE the 1680 cm^{-1} band disappeared completely in 7 h. It may be assumed that the (I) compound, formed during reaction, again reacts with PGE to give (II).

To confirm this assumption, we studied the reaction of (I) [1] with a double mole amount of PGE (200°C, 7 h, Ar). The IR spectrum of the obtained product was identical with the spectrum of 3-phenyl-5-phenoxymethyl-2-oxazolidone [1, 3], except for a more intense absorption band in the 2900-3000 cm^{-1} region, which is characteristic for the CH_2 group, and after purification it melted at 130-132°. Oxazolidone (II) has mp 134.5-136.5° [3]. The observed deviation of the melting point of the obtained product from the literature data for (II) can be explained by its contamination with the linear polymerization product of PGE.



The formation of (II) from (I) and PGE was confirmed by the mass spectrometry method by the presence of peaks at 269, 132, 118, 105, 104, 91, 77, and 51 [5]. In addition, a mass peak at 225 was detected, which cannot be assigned to product (II).

On the basis of the IR and mass spectral data, we show in Scheme 1 a course of the process which can be proposed for the reaction of (I) with PGE.

Phenyl isothiocyanate reacts in a similar manner with an excess of an alkene oxide [6]. However, compound (III) could not be isolated in our case, possibly because (III) is polymerized under the experimental conditions with an opening of the aziridine ring.

EXPERIMENTAL

The IR spectra were taken on UR-20 and Perkin-Elmer 457 instruments, and the mass spectra were taken on an MS-30 instrument (AEI).

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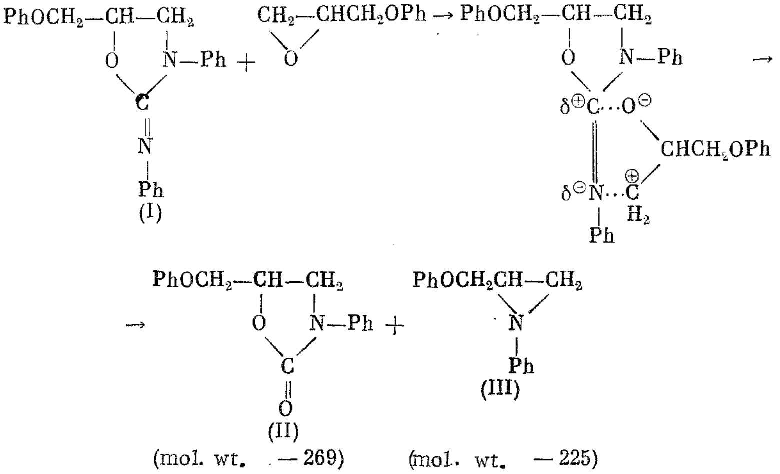
Scheme 1



The reaction of DPC with PGE was run in sealed Pyrex glass ampuls, filled with argon, at 180° for 0.5-7 h. The reaction of DPC with PGE and H_2O, and also of 3-phenyl-5-phenoxymethyl-2-N-phenyliminooxazolidine (I) with PGE, were run under the same conditions. The obtained products were recrystallized from acetone. 3-Phenyl-5-phenoxymethyl-2-oxazolidone (II), mp 130-132°, was obtained in 80% yield.

CONCLUSIONS

3-Phenyl-5-phenoxymethyl-2-N-phenyliminooxazolidine, formed by the reaction of diphenylcarbodiimide with phenyl glycidyl ether, reacts with an excess of the latter to give 3-phenyl-5-phenoxymethyl-2-oxazolidone.

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