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REACTION OF OXAZOLIDONE-2 WITH ETHYLENE OXIDE. XIV*.

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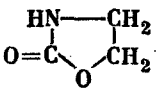
The reaction of ethylene oxide with oxazolidone-2 under various conditions is investigated. A method of preparing 3-β-hydroxyethyloxazolidone-2 from ethylene oxide and oxazolidone-2 under pressure in an autoclave, at 72-75° in aqueous medium, is described.

Mixtures of oxazolidine derivatives [1, 2] are obtained by reacting cyanamide with olefin oxides. These compounds are obtained individually when calcium cyanamide reacts with propylene oxide [3], and ethylene chlorohydrin [4], and when 2-imino-5-methyloxazolidine reacts with propylene oxide [5].

The present communication describes the preparation of 3-β-hydroxyethyloxazolidone-2 from ethylene oxide and oxazolidone-2 (I) (in an autoclave, in aqueous medium, at 72-75°, 4 hr). The yield of 3-β-hydroxyethyloxazolidone-2 (II) (yellowish viscous oil) depended on the temperature and reaction time. At 72-75°, the yield was quantitative, at 45-48° 82.3%, at 25-30° 63%. At 45-50° there was almost no reaction in 2 hr.



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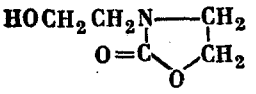

II

A 60% yield of the oxazolidone II was obtained in non-aqueous medium at 70-72° for 4 hr, and there was practically no reaction at a lower temperature. This is explained by the catalytic effect of water on the reactivities of the oxides in reaction with amines [6]. The individuality of the oxazolidone II was proved by its nitrogen content and molecular refraction. Compound II was purified by absorption chromatography, as developed for similar compounds [1].

The structure of II was determined by actual synthesis and alkaline hydrolysis [3], to diethanolamine and carbon dioxide, isolated as barium carbonate. The characteristic frequency of the carbonyl group (1700 cm^{-1}) was detected in the IR spectrum of oxazolidone II.

Experimental

3-β-Hydroxyethyloxazolidone-2 (II). A stirred 1 *l* autoclave was charged with 47.5 g (0.546 mole) I, 114 ml (6.33 mole) water, and 47.5 g (1.09 mole) ethylene oxide, and the charge was constantly stirred and heated at 72-75° for 4 hr. Then the water was distilled off from the reaction products, the residue dissolved in dry methanol, filtered, and the methanol distilled off from the filtrate. Yield: 71 g, d_4^{20} 1.3521, n_D^{20} 1.5196. Found: N 10.30, 10.27%, MR_D 29.46. Calculated for $C_5H_9NO_3$: N 10.68%, MR_D 30.21.

REFERENCES

1. A. E. Kretov and I. S. Matveev, ZhOKh, 30, 1837, 1960.
2. A. E. Kretov and I. S. Matveev, ZhOKh, 31, 2885, 1961.
3. A. E. Kretov and I. S. Matveev, ZhOKh, 32, 471, 1962.
4. I. S. Matveev and A. E. Kretov, ZhPKh, 35, 2106, 1962.
5. I. S. Matveev and A. E. Kretov, ZhOKh, 32, 3220, 1962.
6. K. A. Krasulskii, Investigations into the Reaction of Ammonia and Amines with Organic Oxides [in Russian], Kiev, 10, 1911.
7. N. N. Lebedev and M. M. Smirnov, Izv. vuzov, Khimiya, 3, 104, 1960.
8. I. S. Matveev, ZhOKh, 34, 3795, 1964.

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*For Part XIII see [8].