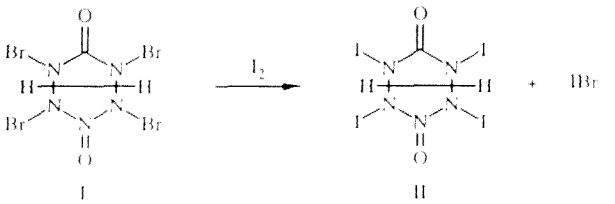
SUCCESSFUL SYNTHESIS OF 2,4,6,8-TETRAIODO-2,4,6,8-TETRAAZABICYCLO[3.3.0]OCTANE-3,7-DIONE

A. Yu. Yagovkin, A. A. Bakibaev, and E. L. Bystritskii

In spite of the fact that the N-iodo derivatives of amides [1] and imides (N-iodosuccinimide [2]) are fairly well known, attempts at the synthesis of the N-iodo derivatives of 2,4,6,8-tetraazabicyclo[3.3.0]octane-3,7-dione (glycoluril) have not led to success, although N-chloro- and N-bromo-substituted glycolurils are easily formed during the reaction of glycoluril with molecular halogen in an aqueous medium [3].

We found that the reaction of tetra-N-bromoglycoluril (I) with molecular iodine in a polar solvent (1,4-dioxane, acetic acid, acetic anhydride) at room temperature gave a high yield of tetra-N-iodoglycoluril (II) and iodine bromide. The analogous reaction of tetra-N-chloroglycoluril with iodine does not give any N-iodo-substituted compounds.



I II

Thus, 4.58 g (0.01 mole) of tetra-N-bromoglycoluril (I) and 10.1 g (0.04 mole) of metallic iodine were stirred in 10 ml of acetic anhydride for 1 h. The precipitated tetraiodide (II) was filtered off and dried, and 5.62 g (87%) of the compound was obtained; the active iodine content (iodonitrile) of compound (II) was 156.1% (calculated 156.8%). The iodine bromide (bp 116°C) was distilled from the mother solution; published data [4], bp 116°C. In addition, we demonstrated qualitatively that N-iodo derivatives were formed during the reaction of other N-bromo derivatives (acetamide, succinimide) with iodine with the release of iodine bromide.

The discovered method for the synthesis of N-iodoamides improves the accessibility of the N-iodo derivatives in organic synthesis, since simple methods are available for the production of N-bromoamides.

The elemental analysis of compound (II) for C, H, and N agreed with the calculated data.

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