REACTION OF TETRAFLUOROHYDRAZINE WITH ALIPHATIC OXIMES

A. V. Fokin, V. I. Zimin, and Yu. N. Studnev

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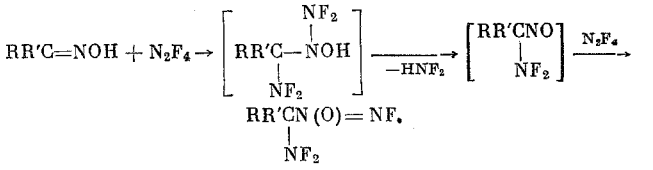
The reaction of tetrafluorohydrazine with p-quinonedioxime leads to the formation of p-bis(N-fluoroazoxy)benzene [1]. This reaction with aliphatic oximes takes a more complicated course and gives difluoroamino-N-fluoroazoxyalkanes. A reaction mechanism can be suggested which is similar to Pilotti's reaction



(I) $R = R' = CH_3$; (II) $R = CH_3$, $R' = C_2H_5$; (III) $R + R' = (CH_2)_5$

The reaction takes place at 60-80°C and a pressure of 1.5-2 atm. In some cases a sudden pressure increase has been observed which has caused breakage of reaction vessels, which might be connected with the radical-chain character of the process.

Substances (I)-(III) are nonviscous colorless liquids with an increased sensitivity towards external influences. The IR spectra of compounds (I)-(III) are in agreement with the data in [2-5] (1520-1510 cm^{-1} band of the N-fluoroazoxy group and 990-830 cm^{-1} band of the N–F bond).

1-Difluoroamino-1-N-fluoroazoxycyclohexane (III). 7.4 g cyclohexanoneoxime in 40 ml ethyl acetate and 6.4 g tetrafluorohydrazine were placed in an evacuated glass reaction vessel. The vessel was kept at 70-75° for 8 h and allowed to stand overnight. Yield 3.1 g (III), bp 46° (1 mm), n_D^{20} 1.4535, d_4^{20} 1.291. Found %: F 28.14, N 21.63. $C_6H_{10}F_3N_3O$. Theory %: F 28.93, N 21.32.

The data for the other compounds were: (I) bp 54° (30 mm), n_D^{20} 1.3952, d_4^{20} 1.324. Found %: F 36.74, N 26.55. $C_3H_6F_3N_3O$. Theory %: F 36.31, N 26.75. (II) bp 74 (50 mm), n_D^{20} 1.4073, d_4^{20} 1.285. Found %: F 32.91, N 23.87. $C_4H_8F_3N_3O$. Theory %: F 33.33, N 24.56.

LITERATURE CITED

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