## REPLACEMENT OF THE CHLORINE ATOMS IN

DICHLOROFORMAL BY FLUORINE ATOMS

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In our recent work [1], we proposed a method for the preparation of aliphatic dichloroformals, in which the chlorine atoms cannot be replaced by fluorine using the generally known methods with retention of the molecular structure.

Use of antimony trifluoride or pentafluoride in sulfuryl chloride in the presence of catalytic amounts of bromine permitted the replacement of the chlorine atoms in bis(2,2-difluoro-2-nitroethyl)- (I) and bis(2-fluoro-2,2-dinitroethyl)dichloroformal (II) by fluorine. This method is general for dichloroformals with electron-withdrawing substituents (dichloroformals with electron-donor substituents are unknown) and gives difluoro-formals (III) and (IV) in 70% yield.

 $\begin{array}{ll} Cl_2C(OCH_2R)_2 & \frac{SbF_3 & \text{or } SbF_5}{SO_2Cl_2} & F_2C(OCH_2R)_2 \\ (I), (II) & (III), (IV) \\ R = CF_2NO_2 & (I), (III); & CF(NO_2)_2 & (II), (IV) \end{array}$ 

A sample of 0.01 mole  $\rm SbF_3$  and 0.01 g bromine were added with stirring to a solution of 0.01 mole (I) or (II) in 10 ml  $\rm SO_2Cl_2$  at 20-25°C. The reaction mixture was stirred for 24 h. The volatile compounds were removed using a water pump and 20 ml  $\rm CH_2Cl_2$  was added. The solution was washed with water, dried over MgSO<sub>4</sub> and evaporated. Vacuum distillation gave bis(2,2-difluoro-2-nitroethyl)difluoroformal (III), bp 109-110°C (18 mm), np<sup>20</sup> 1.363 and bis(2-fluoro-2,2-dinitroethyl)difluoroformal (IV), bp 80-81°C (1 mm), np<sup>25</sup> 1.4134 [2].

## LITERATURE CITED

- 1. L. T. Eremenko, G. V. Oreshko, and M. A. Fadeev, Izv. Akad. Nauk SSSR, Ser. Khim., 2636 (1986).
- 2. H. M. Peters and R. L. Simon, US Patent No. 3,922,311 (1975).

Division of the Institute of Chemical Physics, Academy of Sciences of the USSR, Chernogolovka. Translated from Izvestiya Akademii Nauk SSSR, Seriya Khimicheskaya, No. 6, pp. 1439-1440, June, 1987. Original article submitted April 7, 1987.