

NUCLEOPHILIC ISOMERISATION OF HEXAFLUOROPENE OXIDE

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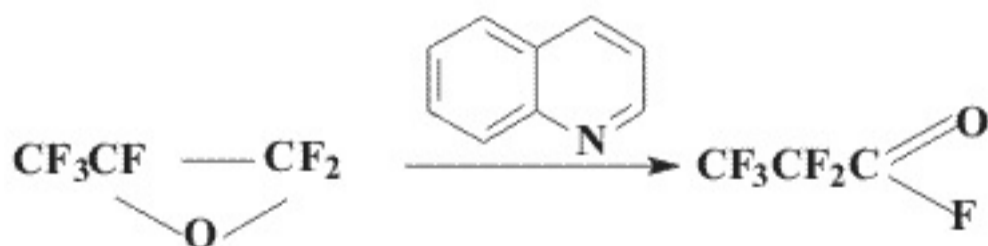
Attempts to obtain pentafluoropropionyl fluoride (PPF) undertaken earlier [1-8], except for the synthesis method of this compound by the action of Yarovenko reagent on pentafluoropropionic acid [9], have no preparative importance.

With the goal to develop a convenient method to obtain PPF in the present work we studied "nucleophilic" isomerization of hexafluoropropene oxide (HFPO). The latter is an available product of fluoroorganic manufacture.

We studied the action of various reagents (KF, CsF, pyridine, triethylamine and other bases) on HFPO both in a flow system by passing through a heated pipe filled with fluorides of alkali metals and by bubbling HFPO through a base solutions (pyridine, triethylamine, quinoline) followed by capturing the forming products.

As the result of these experiments it has been found that variations of HFPO flow rate, temperature, time of interaction and other process parameters do not allow to attain full conversion of HFPO into PPF and as the boiling temperatures of these substances are very close (~ -28 o C) it is impossible to isolate PPF in pure form.

At the same time it was found that full HFPO isomerization takes place in a close system under the action of quinoline.



The found method is extremely convenient and allows producing PPF in great quantities.

Experimental

Quinoline (200g, 1.55 mol) and hexafluoropropylene oxide (2400g, 14.46 mol) are placed in a steel autoclave (2L water capacity) with a needle valve cooled to a temperature of -70 °C. The autoclave is hermetically sealed and heated. At a temperature of 10 °C there is observed a temperature jump up to 60 °C in the autoclave.

The autoclave is placed in a rocking furnace, the temperature is raised to 100 °C and kept for 2 hours. Then the autoclave is cooled to room temperature, the product is collected

through the needle valve in a trap cooled with dry ice. There was obtained 2200g of Hexafluoropropionyl fluoride of 97% purity in 92% yield, BP=-28°C.

^{19}F NMR: 8.0 ppm (CF_3), 46 ppm (CF_2), -98 ppm (COF)

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