

NUCLEOPHILIC ISOMERISATION OF HEXAFLUORO

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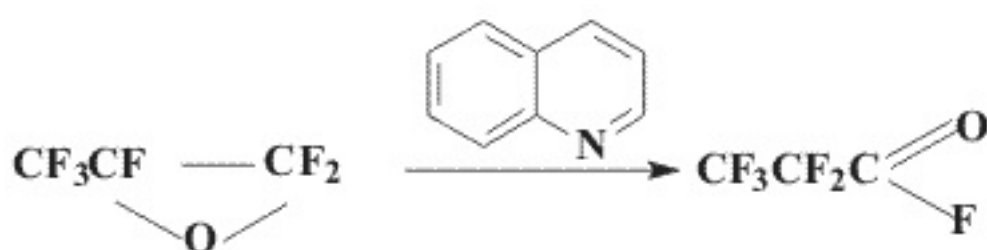
Attempts to obtain pentafluoropropionyl fluoride (PPF) undertaken earlier [1-8] , except for this compound by the action of Yarovenko reagent on pentafluoropropionic acid [9], have no preparative value.

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

We studied the action of various reagents (KF, CsF, pyridine, triethylamine and other bases) by passing through a heated pipe filled with fluorides of alkali metals and by bubbling HFPO through solutions of 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 and other process parameters do not allow to attain full conversion of HFPO into PPF and as the boiling points of these substances are very close (~-28 °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 closed system under pressure.



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 an autoclave (100 L capacity) with a needle valve cooled to a temperature of -70 °C. The autoclave is hermetically sealed. 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. After cooling to room temperature, the product is collected through the needle valve in a trap cooled to -78 °C. 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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