Fluorine-containing pyrazoles

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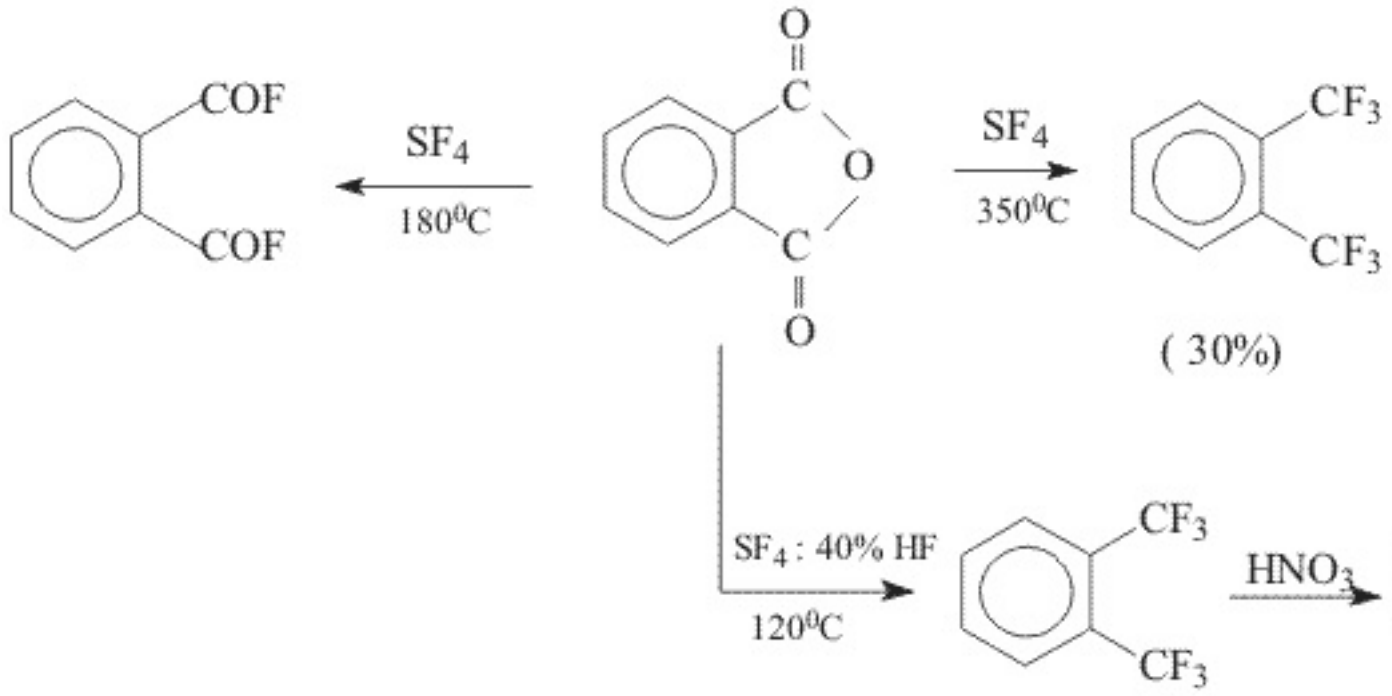
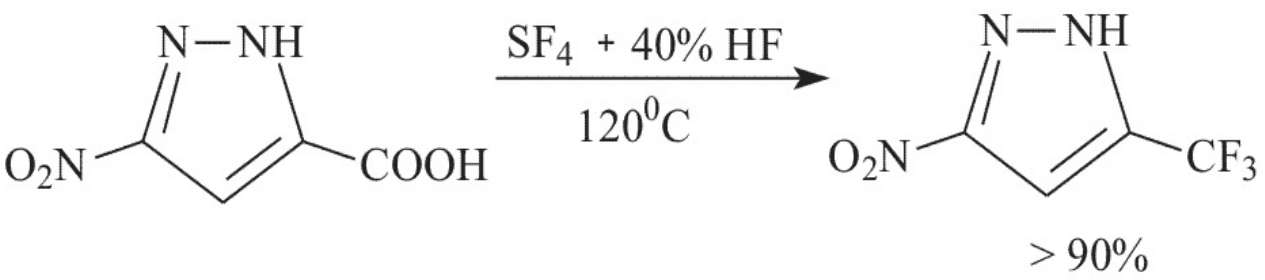
It has been shown earlier that for production of 1,2-bis (trifluoromethyl) benzene from phtali 350 ^{o}C is necessary [1,2].



During the process of development of a method to produce 2,3-bis(trifluoromethyl)nit considerable decrease in the reaction temperature by addition of a small quantity of hydroflu H_2O) into the reaction mixture.

It turned out that the use of SF_4 with addition of hydrofluoric acid could be successfully used in pyrazoles. Thus, by treatment of 3-carboxy-5-nitropyrazole with this mixture a co trifluoromethyl group was synthesized in a yield up to 90%.



Experimental

1,2-Bis(trifluoromethyl)benzene

Phtalic anhydride (45g, 0.3 mol) and hydrofluoric acid (7mL) were placed in a steel autocla autoclave was cooled with dry carbonic acid at first and then with liquid nitrogen, then 85 mL mol) preliminary condensed were poured from a trap. The autoclave was hermetically sealed heated at a temperature of 120^{o}C for 10 hours. Next day the autoclave contents was poure added, washed with water , dried with $MgSO_4$ and after distillation from CH_2Cl_2 the residue v produced 35g (61%) of 1,2-bis(trifluoromethyl)benzene, Tb= 420/15 mm Hg(compare [2]).

3-trifluoromethyl-5-nitropyrazole

Similar to the above experiment from 3-carboxy-5-nitropyrazole (80g, 0.5 mol), hydrofluoric tetrafluoride (216g, 2.0mol) there was produced 83g of the product (90%), Melting Point= 65°

Found,%: C, 26.32; H, 3.40; N, 22.42 C4H6N3O4,
Calculated,%: C, 26.08; H, 3.26; N, 22.82. NMR: 'SF4.2 ppm. (CF_3)

References

1. A.P.Khardin, B.N.Gorbunov, P.A.Protopopov, Chemistry of sulfur tetrafluoride, Ed. Saratov U

2. W.R. Hasek, W.C. Smilh., V.A. Engelhardt . I. Aw. Chem. Soc.,82,543 (1960)