Preparation of 1-oxo, 4-chlorobutyloferrocene; description of hexanitrohexaazaisowurtzitane (CL-20) friction sensitivity

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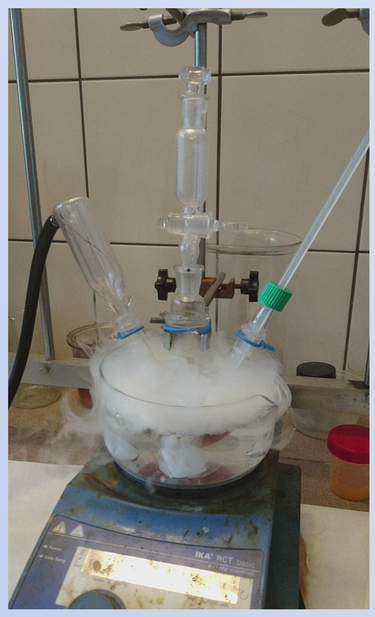
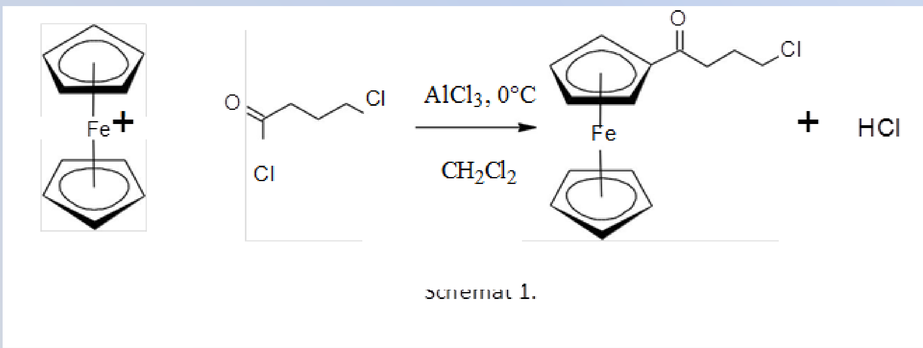


1st stage : Preparation of 1-oxo, 4-chlorobutyloferrocene

Aim: Preparation of 1-oxo, 4-chlorobutyloferrocene

Preparation of 1-oxo, 4-chlorobutyloferrocene is the first stage of butacene synthesis. Butacene is a compound that stabilises combustion speed of solid rocket fuel and prevents inhomogeneous burning.



Schemat 1.

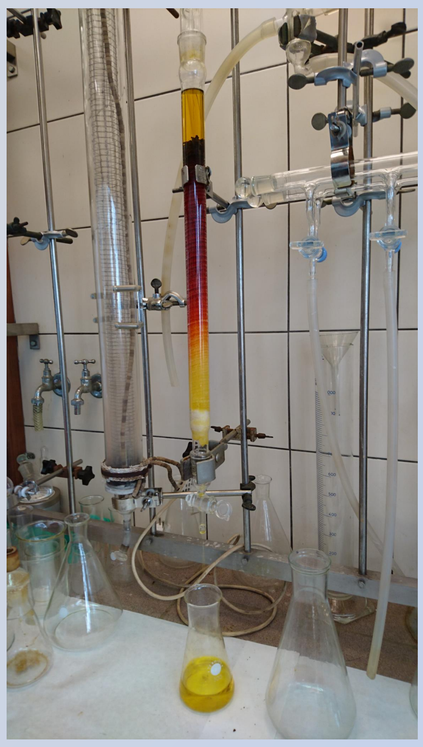
Method

1) Drying molecular sieves (type A3)
2) Drying the dissolvent (Dichloromethane) over molecular sieves
3) Dissolution of ferrocene in the dry dissolvent
4) The reaction in a three-necked flask in a water and oxygen-free conditions: addition of 4-chlorobutyryl chloride; addition of catalyser $AlCl_3$; change in the colour of the mixture from brown to navy blue
5) Addition of cold water – hydrolysis of $AlCl_3$
6) Transfer of the whole mixture into a separating funnel. Rinsing the organic phase with water, NaOH, HCl. The by-products were washed out.
7) Drying the mixture over $MgSO_4$ (dry)
8) Filtration of water-free mixture
9) Passing the mixture through a silica gel column, using a mixture of hexane and ethyl acetate in a ratio 9:1. The first formed yellow fraction was unreacted ferrocene. After using a mixture of hexane and ethyl acetate in ratio 4:1 the first formed fraction (red) was the product, the fraction on the top of the column (black) was unreacted tar.
10) Concentration of the formed product with evaporator

Result: 2.66 g of the product

Analysis - Infrared spectroscopy confirmed that the obtained product is 1-oxo, 4-chlorobutyloferrocene.

The reaction was based on a recipe.



2nd stage: Description of Hexanitrohexaazaisowurtzitane Explosives friction sensitivty

Aim: Description of Hexanitrohexaazaisowurtzitane

Method: Peters apparatus measuring the friction force at which the CL-20 never explodes (upper value of insensitivity) and the friction force at which CL-20 always explodes (lower sensitivity value).

Result: The upper value of insensitivity of CL-20 is 248.4 N. The lower sensitivity value is 356.4 N. Thus, when a specimen contains CL-20, to work with it safely, one should apply friction strength lower than 248 N. If one needs the specimen to explode, he should apply friction strength of at least 356,4 N.