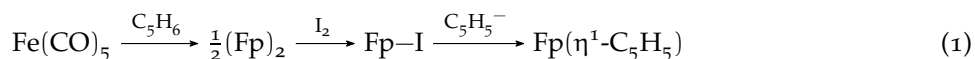


Ring Whizzer Lab

Overview

In this project, you will prepare an iron cyclopentadienyl compound with a fluxional structure, that is, a structure that is constantly changing. In this case, the η^1 ligand is spinning such that which carbon is σ bound to the Fe rapidly changes — rapidly enough that all five carbons of that ring appear equivalent by NMR, as also do all five protons.

The preparation is shown in Reaction 1. “Fp” (pronounced “fip”) is short for $\text{Fe}(\text{CO})_2(\eta^5\text{-C}_5\text{H}_5)$, and “ η^5 ” (read “eta-five” or “pentahapto”) indicates that five carbons of the ligand are attached to the metal. In older papers, these are also referred to as “sigma” and “pi,” depending whether there is only a σ -bond (η^1) or also π - (or δ -) bonding between the carbon fragment and the metal.



You will want to start by cracking dicyclopentadiene, so that you’ll have cyclopentadiene for both the first step and to make the anion used in the third step. Since cyclopentadiene reverts back to dicyclopentadiene, it is best to store it in the freezer once you’ve cracked it. Plan to crack 1.5 times what you’ll need.

The compounds in this preparation are somewhat air-sensitive, and hence need to be prepared under nitrogen, using Schlenk techniques. See either Dr. Trujillo or Dr. Youmans for instruction.

References

The Hallam/Pauson and Piper/Wilkinson articles below were obtained by interlibrary loan, hence copies are included in this directory. The rest may be obtained on-line or at the library.

Cracking Dicyclopentadiene:

Any standard organic lab text will contain this — Pavia, Williamson, Vogel, etc.

First step:

King, R.B.; Stone, F.G.A. *Inorg. Synth.* **1963**, 7, 110-2.

Hallam, B.F.; Pauson, P.L. *J. Chem. Soc.* **1956**, 3030-7.

- Both of these are on a huge scale. Cut your preparation down to something reasonable, eg. 5 g iron pentacarbonyl.
- Using cyclopentadiene in a pressure bottle seems to work better than dicyclopentadiene at 1 atm.
- Flush the pressure bottle with N_2 before introducing the iron carbonyl and cyclopentadiene, rather than dealing with CO_2 .

Second step:

King/*Inorg. Synth.* monograph above.

Third Step:

Piper, T.S.; Wilkinson, G.J. *Inorg. Nucl. Chem.* **1955**, 3, 104-124. (print at 85% ?)

- Perhaps best to omit the sublimation of the evaporation residue? Otherwise, be sure to use very gentle heat and a good vacuum (< 1 mmHg).

Context and further information:

Bennett Jr., M.J., et al. *J. Am. Chem. Soc.* **1966**, 88, 4371-6.

Cotton, F.A. *Acc. Chem. Res.* **1968**, 1, 257-65.