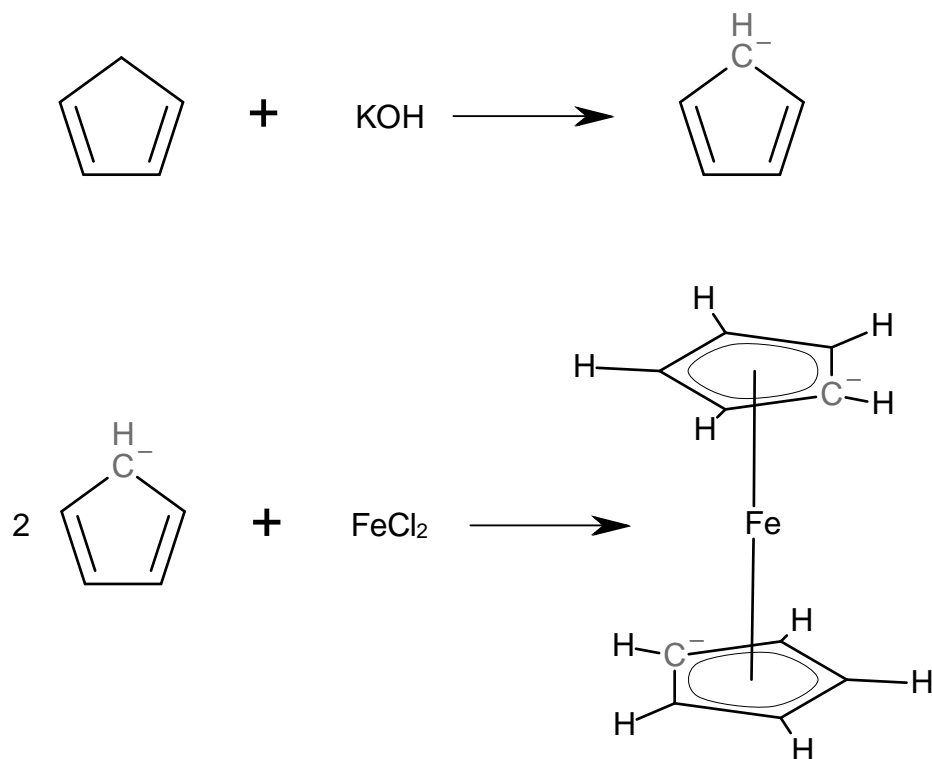


# Ferrocene

Procedure developed and written by Anthony Winston

## 1. Reaction



## 2. Procedure

Set up a 10 mL round bottom flask fitted with a screw cap, septum, and two needles inserted. Grind some KOH in a mortar and add to the flask 750 mg of the powdered KOH and then 1.25 mL of dimethoxyethane (DME). Add 300  $\mu$ L (240 mg, 3.6 mmoles) of freshly prepared cyclopentadiene, close the flask, and flush briefly with nitrogen. Shake occasionally while preparing the ferrous chloride solution.

To a 3 mL conical vial fitted with a cap, septum with two needles, and a spin vane, add 350 mg (1.76 mmoles) of FeCl<sub>2</sub>·4H<sub>2</sub>O and 1.5 mL of DMSO. Flush with nitrogen and stir to dissolve the ferrous chloride. When the ferrous chloride has mostly dissolved, start transferring the iron solution to the cyclopentadiene-KOH mixture by means of a syringe. Add the FeCl<sub>2</sub> solution gradually over a period of about 10 minutes with shaking after each addition. When the solution has all been transferred, shake intermittently for another 10 minutes. Open the reaction flask and add the reaction mixture to about 10 mL of dilute HCl containing some ice. Add concentrated HCl, with stirring, until the mixture is strongly acidic. Acidification causes iron oxides to dissolve. Collect the orange solid by filtration using the Hirsh funnel, wash with water, and allow the solid to dry. Sublime about 50 mg using the vacuum sublimation apparatus at a block temperature of 100-120°C. Use 100 mg of the crude product to prepare mono- and diacetylferrocene.

### 3. Report: Ferrocene

Name \_\_\_\_\_ Date \_\_\_\_\_ Exp. No. \_\_\_\_\_

Chemical Reaction:

#### Reagents:

Compound	Mol Wt	Density	Wt, mg	Vol, L	Mmole

#### Products:

Compound	Mol Wt	Density	Wt, mg theoretical	Vol. $\mu$ L Theoretical	Mmol Theoretical

Attach IR spectrum, Gas Chromatograms, etc.

Yield \_\_\_\_\_ mg \_\_\_\_\_ mmol

Yield \_\_\_\_\_

Mp or bp range \_\_\_\_\_, Lit. \_\_\_\_\_

Evidence for confirmation of structure

IR Bands and Assignments