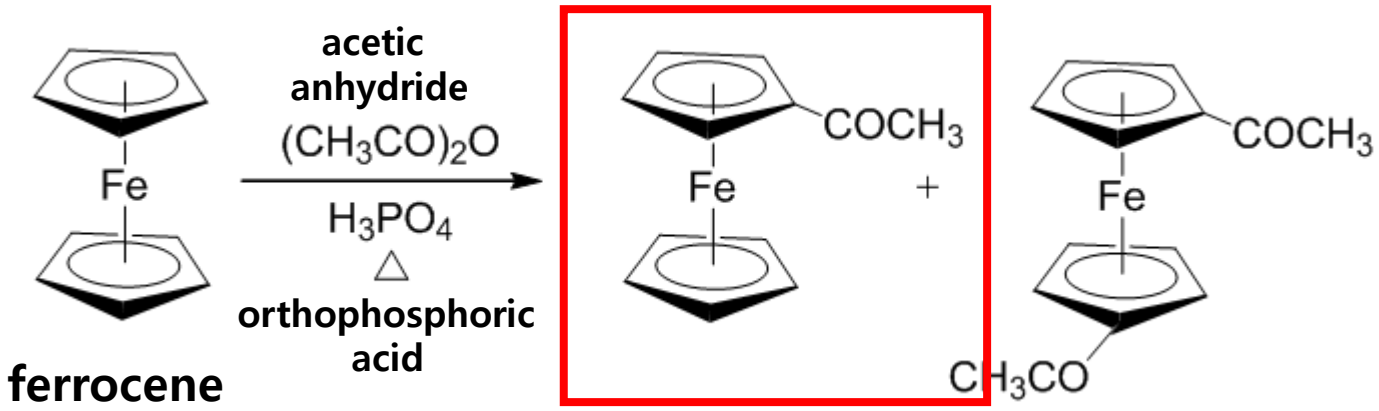
Acetylation of ferrocene and reduction of acetylferrocene

Synthetic scheme



Acetylferrocene is the organoiron compound. It consists of ferrocene substituted by an acetyl group on one of the cyclopentadienyl rings.

Acetylation

1. Add ferrocene (1.5 g) to acetic anhydride (5 mL) in 25 mL of a round bottom flask.
2. Add orthophosphoric acid (1 mL) dropwise.
3. Stir the mixture for 20 minutes in a boiling water bath.

   * You should set a thermometer.
4. Pour the hot mixture onto crushed ice (half of 250 mL beaker). (Remained organic product in round bottom flask have to be washed out water.) * Hot! So be careful when you handle the glasswares and clamps.

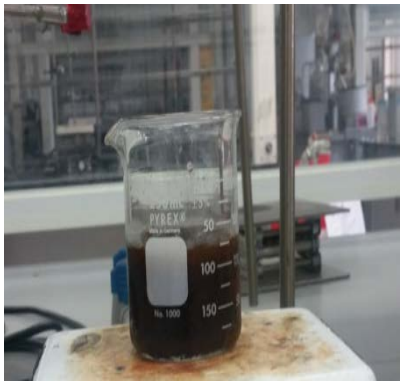



Acetylation

5. Add 20~30 mL of saturated sodium bicarbonate solution.

*Caution: The saturated solution should be added dropwise and shake gently with a hand.

6. Cool the neutralized mixture in an ice bath for 5 minutes.
7. Filter off the brownish yellow solid.
8. Wash the product with petroleum ether.
9. All volatiles are removed by rotary evaporator.
10. Weigh your product and calculate the percent yield (%).



Reduction of Acetylferrocene

1. Add acetylferrocene (0.5 g) to a 50 mL of Erlenmeyer flask and dissolve in ethanol (10 mL).
2. Dissolve $NaBH_4$ (0.4 g) in water (2 mL)

   and add $NaBH_4$ solution dropwise to the acetylferrocene solution.
3. Stir the reaction mixture for 5 minutes at room temperature.

Reduction of Acetylferrocene

4. Add distilled water (20 mL) and transfer to a separate funnel
5. The organic product was extracted with diethylether (20 mL X 2).
6. Dry the ether solution over magnesium sulfate and the precipitate is filtered by filter paper.

Reduction of Acetylferrocene

6. Remove all volatiles using rotary evaporator.
7. Weigh your product and calculate the percent yield (%).

To do (Minimum)

- Prepare two NMR samples (Acetylferrocene, reduction of acetylferrocene)
- Analyze of NMR
- Draw the reaction mechanism and explain the pathway of acetylation and reduction steps.
- Explain why the acetylation of ferrocene proceeds faster in ethanol than in benzene solution.

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