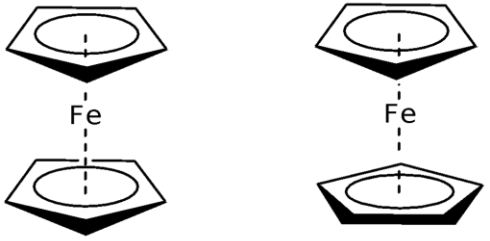
Experiment 3

Preparation of Ferrocene

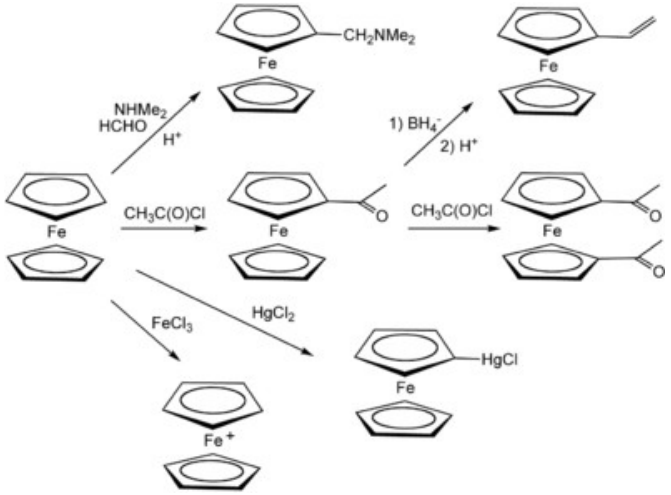
Background Knowledge

- Ferrocene



Eclipsed Staggered

Properties	
Molecular formula	$C_{10}H_{10}Fe$
Molar mass	186.04 g/mol
Appearance	light orange powder
Density	1.107 g/cm³ (0°C), 1.490 g/cm³ (20 °C)[1]
Melting point	172.5 °C[2]
Boiling point	249 °C
Solubility in water	Insoluble in water, soluble in most organic solvents



Rapid growth of organometallic chemistry is often attributed to the excitement arising from the discovery of ferrocene and its various a nalogues

- Chemical Equation

$2\ NaC_5H_5 + FeCl_2 \rightarrow Fe(C_5H_5)_2 + 2\ NaCl$

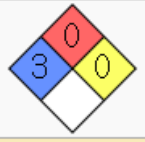
$FeCl_2.4H_2O + 2\ C_5H_6 + 2\ KOH \rightarrow Fe(C_5H_5)_2 + 2\ KCl + 6\ H_2O$

Chemicals

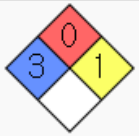
- Diethyl ether

Hazards	
MSDS	External MSDS
R-phrases	R12 R19 R20/22 R66 R67
S-phrases	S9 S16 S29 S33
Main hazards	Extremely Flammable, harmful to skin
NFPA 704	
Flash point	−45 °C[1]
Autoignition temperature	160 °C[1]
Explosive limits	1.9–48.0% [2]

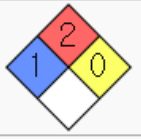
- $FeCl_2$

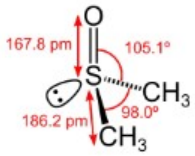
Hazards	
NFPA 704	

- KOH

Hazards	
MSDS	ICSC 0357
GHS pictograms	 [5]
GHS signal word	Danger
GHS hazard statements	H302, H314[5]
GHS precautionary statements	P280, P305+351+338, P310[5]
EU Index	019-002-00-8
EU classification	 C Xn
R-phrases	R22, R35
S-phrases	(S1/2), S26, S36/37/39, S45
NFPA 704	
Flash point	Non-flammable
LD_{50}	273 mg/kg (oral, rat)[6]

- DMSO

Hazards	
MSDS	External MSDS
R-phrases	R36/37/38
S-phrases	S26, S37/39
Main hazards	Irritant and flammable
NFPA 704	
Flash point	89 °C



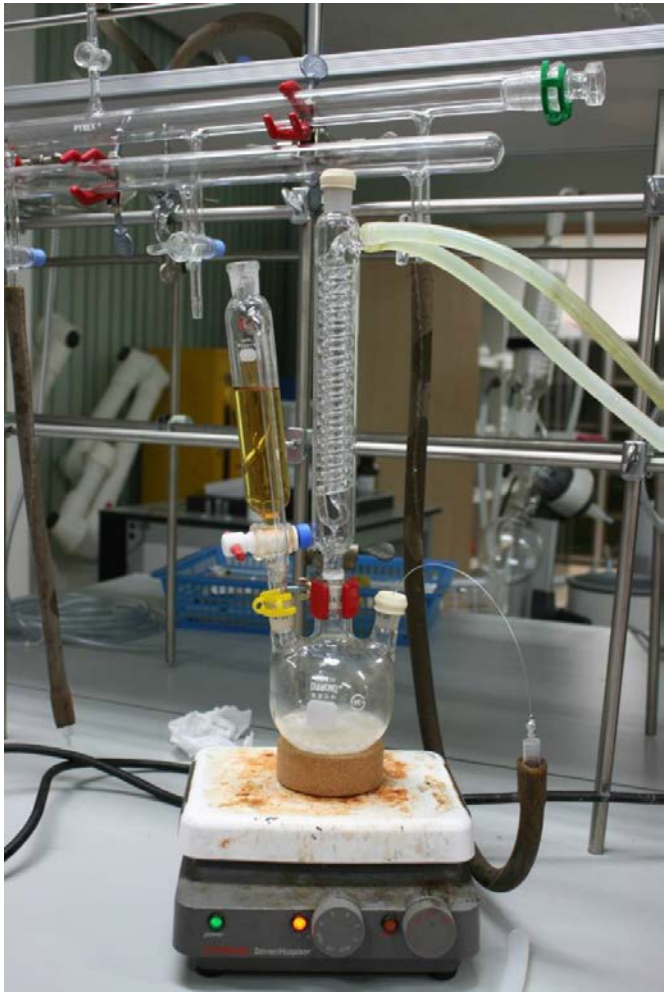
Preparation of Ferrocene (by TA)

① Check that the flask contains at least 100 ml of dicyclopentadiene.

② Collect the distillate that condenses in the range 42 ~ 44 ℃ and keep it cooled with an ice bath around the receiving flask.

③ Store the product in a refrigerator at low temperature

Preparation of Ferrocene



① Fix a 250 ml three neck flask with a mechanical stirrer to a Liebig condenser.

② Fix a 50 ml dropping funnel to one side neck by means of a side-arm adaptor.

③ Connect a bubbler to the other side to prevent air entry and monitor the nitrogen flow rate.

Preparation of Ferrocene

④ Charge the flask with diethyl ether (50 ml) and flake potassium hydroxide (20 g), stir well and flow the nitrogen gas.

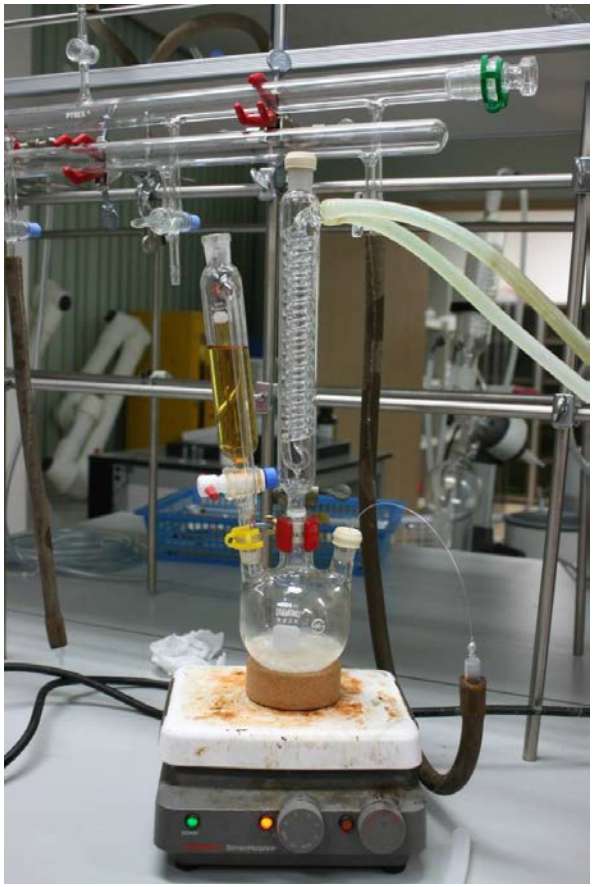
⑤ Meanwhile, dissolve finely powdered iron(II) chloride tetrahydrate (5 g) with dimethylsulphoxide in 50 ml round bottom flask, degassed by bubbling nitrogen through it (20 ml, avoid skin contact; stirring for an hour).

⑥ Add 4.25 ml cyclopentadiene to the KOH/ether mixture by using a 10 ml syringe.

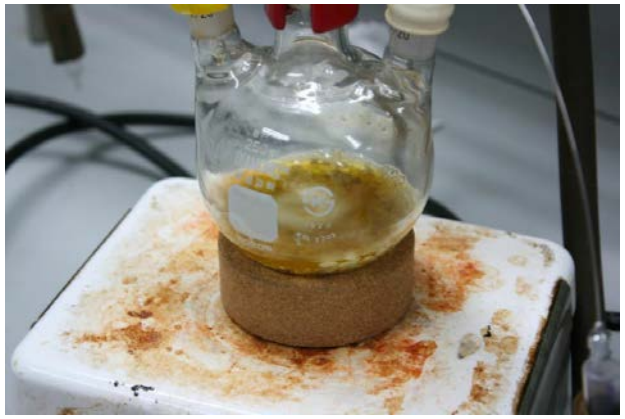
⑦ After 15 min, discontinue the nitrogen flow
and dropwise the iron (II) chloride solution.

⑧ Restore a slow nitrogen flow
and replace any ether lost by evaporation.

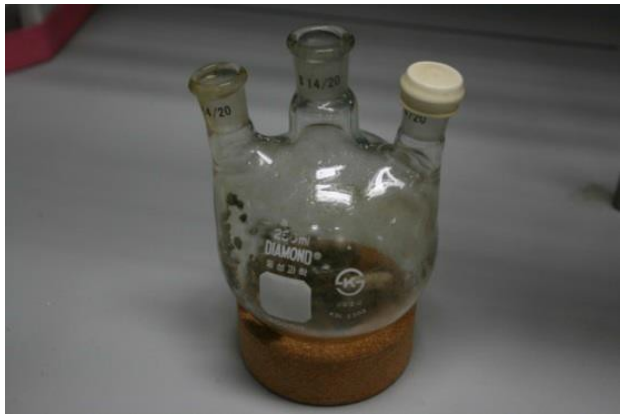
(Care : the KOH/solvent mixture is extremely corrosive)



Preparation of Ferrocene



⑨ Continue stirring for a further 30 min.



⑩ Wash the dark residue with 25 ml ether and transfer it to a separation funnel.

Preparation of Ferrocene

⑪ Wash it with 2 M HCl (2×20 ml)
, distilled water (2×20 ml).

⑫ Extract the ether layer.

(Because ether may boil, extract the solution carefully.)

Preparation of Ferrocene

Preparation of Ferrocene

⑬ Add $MgSO_4$ sufficiently to the solution and wait for 10 min.

⑭ Filter $MgSO_4$ and carefully evaporate off the ether to deposit orange crystals of ferrocene.

Preparation of Ferrocene

⑮ Examine the solubility of ferrocene. (Water, Dichloromethane, Toluene)
Account for your observations in terms of structure and bonding of the mol
ecule

Add ferrocene (0.1 g) to water (5 ml) followed by concentrated nitric acid (5 ml)
– Extreme caution !
Shake the tube gently for 2 min and record your observations

Sublimation and NMR test by TA

To do..

1. Draw d-orbital splitting of Fe in ferrocene and assign each orbital.
2. Explain the reason why cyclopentadiene monomer have to be storaged at low temperature.
3. Why do we use KOH ?
4. Analyze UV & NMR data.
5. More than two about application of ferrocene.