

# Inorganic Chemistry

## Experiment 1

### Iron abstraction from used cans

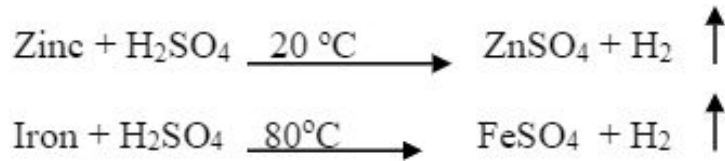
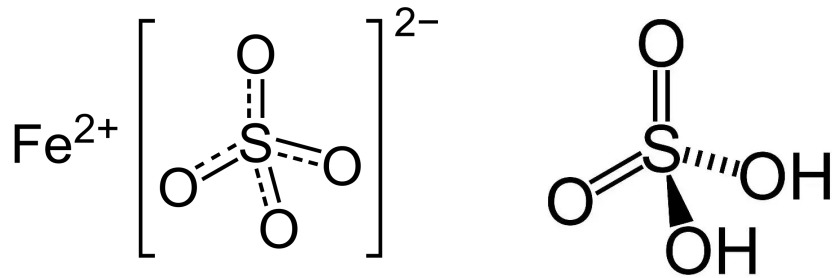
Max Shi, Jeremy Meyerberg, Leigha Tierney

I pledge my honor that I have abided by the Stevens Honor System.

# Purpose

The purpose of this experiment is to demonstrate how iron can be isolated from cans comprised of an alloy of iron and zinc. This experiment also shows the formation of ferrous sulfate hydrated and their ammonia complex

Drawing of structure of the main compound or balanced chemical equation if synthesis is performed



# Reagents and Major Product

Name	M.W. (g/mol)	Density (g/cm <sup>3</sup> )	Amount (ml or g)	Moles	Hazards/Precautions	Role of Reagent
Iron (II) Sulfate $\text{FeSO}_4$	161.91	3.65			Harmful if swallowed. Can cause skin and eye irritation.	Intermediate
Sulfuric Acid $\text{H}_2\text{SO}_4$	98.08	1.83	41mL of 3.0M solution	0.12	Corrosive. Causes eye and skin burns. Also internal and respiratory burns. Handle in a fume hood and with gloves.	Reactant
Potassium Thiocyanate KSCN	97.18	1.89	2.0 mL of 1.0M solution	0.002	Harmful if swallowed, inhaled, or absorbed through skin. Toxic gas is produced when in contact with acids.	Reactant
$\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$ Mohr's Salt	392.21	1.86				Product
Zinc (II) Sulfate $\text{ZnSO}_4$	161.47	3.54			Causes serious eye damage and is harmful if swallowed. Very toxic to aquatic life, dispose properly.	Product
Iron (III) Hydroxide Sulfate $\text{Fe}(\text{OH})\text{SO}_4$	168.92					Byproduct

# Procedures

## Part 1: Pretreatment of used cans

- Cut a piece (about 5 g) of metal from used cans and put them into a 50 ml beaker with 10 ml 3.0 M  $\text{H}_2\text{SO}_4$ .
- Place the baker in air flow hood and observe the hydrogen release and the metal color change.
- As all zinc in the can wreckages are consumed (how can you know?), solution is removed and the left metal wreckages are extensively washed with water.
- Weight the left iron after absorbing all water on the iron surface.

## Part 2: Preparation of $\text{FeSO}_4$

- Put pretreated iron piece into a conical flask (with scale markers) containing 30 ml 3.0 M  $\text{H}_2\text{SO}_4$  solution.
- Place the beaker onto 80°C water bath and observe the reaction.
- Keep adding water to maintain the volume and to avoid  $\text{FeSO}_4$  precipitation. Once the reaction stops (how can you know?), transfer the solutions into a filter and perform vacuum filtration immediately.
- Unreacted iron are washed and weighted.
- Disperse obtained solution into two small conical flasks. Heat one solution on the 60 °C water bath until a crystal membrane is formed on the surface of solution. Move the solution out of water both and allow it cool down to the room temperature. Recover formed crystal, weight, and calculate the yield.

# Procedures (cont.)

## Part 3: Preparation of $(\text{NH}_4)_2\text{SO}_4 \cdot \text{FeSO}_4$ complex

- Add saturated  $(\text{NH}_4)_2\text{SO}_4$  solution into another conical flask with  $\text{FeSO}_4$  solution [ $(\text{NH}_4)_2\text{SO}_4:\text{Fe}=2.2/1.0$ ].
- Place the mixture on a water bath (60~80 °C) for evaporation.
- Remove container out of water bath once crystal membrane is formed on solution surface or solution becomes slight turbidity.
- Leave the solution at room temperature for two hours. Recover  $(\text{NH}_4)_2\text{SO}_4 \cdot \text{FeSO}_4 \cdot 6\text{H}_2\text{O}$  crystal, weight, and calculate the yield.

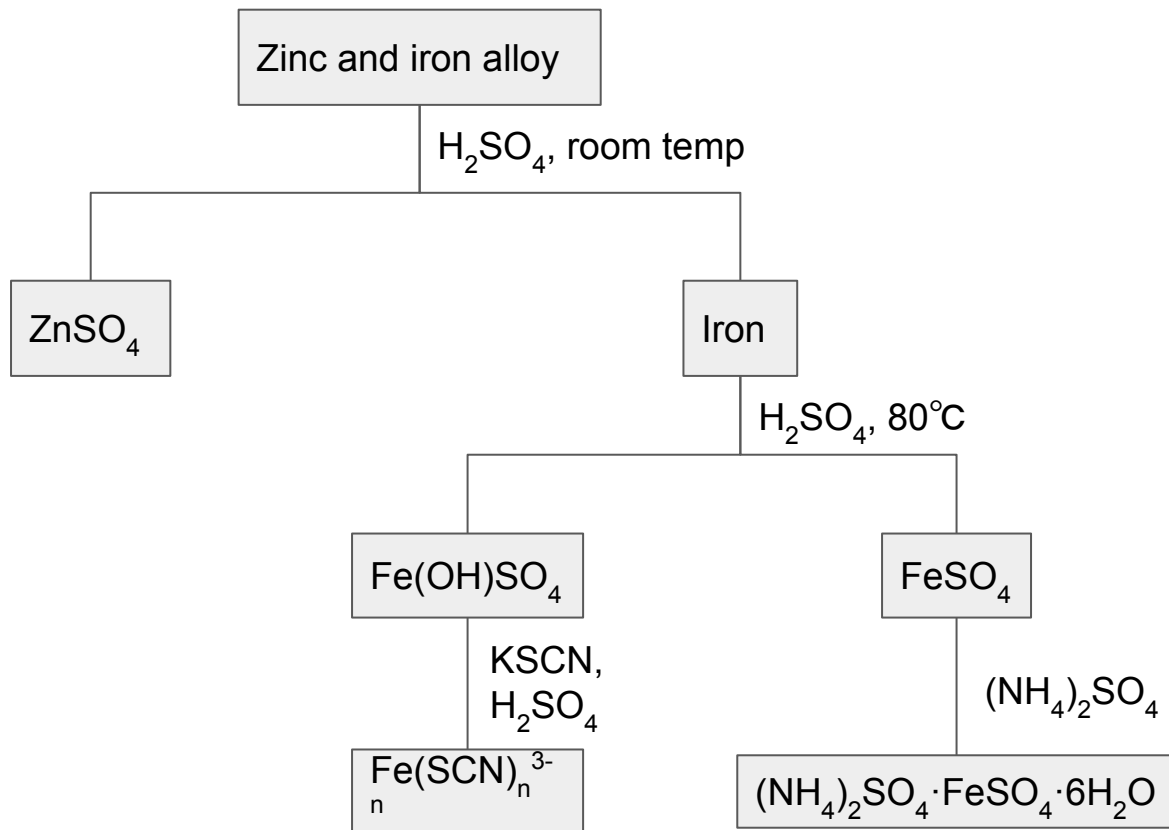
## Part 4: Measurement of trace amount of $\text{Fe}^{3+}$ in $(\text{NH}_4)_2\text{SO}_4 \cdot \text{FeSO}_4$ crystal

- The impurity of  $(\text{NH}_4)_2\text{SO}_4 \cdot \text{FeSO}_4 \cdot 6\text{H}_2\text{O}$  crystal comes from co-precipitated  $\text{Fe}(\text{OH})\text{SO}_4$ .
- $\text{Fe}^{3+}$  in the final product can be measured by KSCN based on the following reaction:



- Dissolve 1.0 g of  $(\text{NH}_4)_2\text{SO}_4 \cdot \text{FeSO}_4 \cdot 6\text{H}_2\text{O}$  complex in small amount of oxygen-free water (10 ml).
- Add 1.0 ml  $\text{H}_2\text{SO}_4$  (3.0 M) and 2.0 ml KSCN solution (1.0M).
- Transfer solution into a 25 ml volumetric flask. Add more oxygen-free water to 25 ml. Transfer 5.0ml prepared solution into 10ml test tube.
- Compare your solution color against the standard solution and estimate the  $\text{Fe}^{3+}$  concentration in your prepared solution.

# Flowchart



# 1. Pretreatment of used cans



# Stepwise Procedure

1. Cut a piece (5g) of metal from used cans and put into a 50 mL beaker with 10 mL  $\text{H}_2\text{SO}_4$ .



2. Place beaker in air flow hood and observe hydrogen release and metal color change

10 mL 3.0 M

5 grams



Stops bubbling

3. Remove solution after reaction is complete, wash leftover metal and weigh after drying.



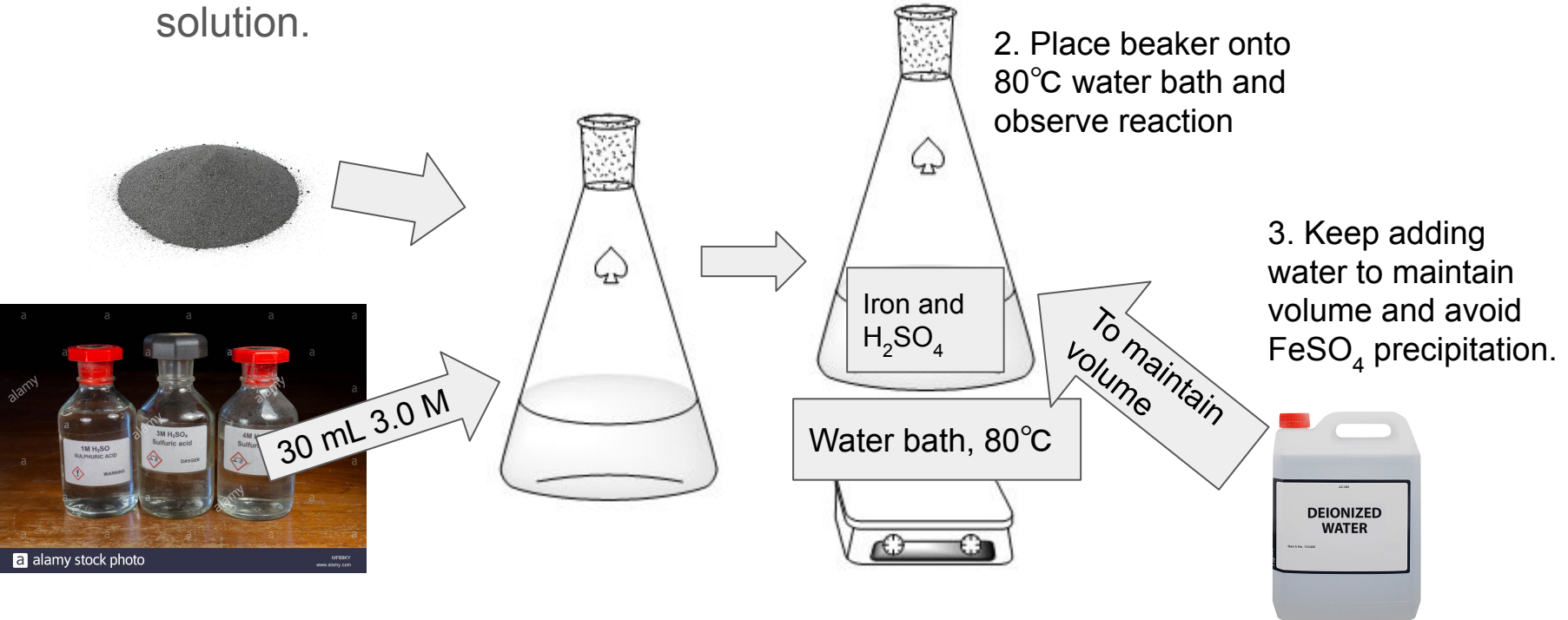
Remove and wash and dry thoroughly, weigh

Iron and zinc alloy

## 2. Preparation of FeSO<sub>4</sub>

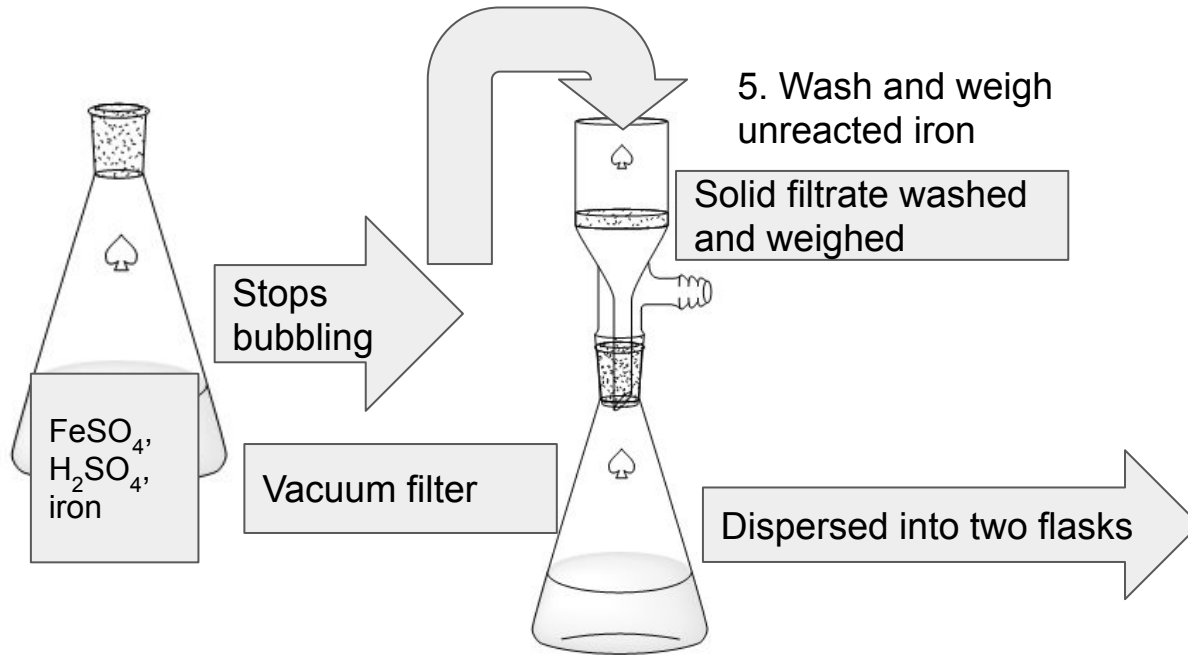
# Stepwise procedure

1. Put pretreated iron piece into conical flask containing 30 mL 3.0 M  $\text{H}_2\text{SO}_4$  solution.

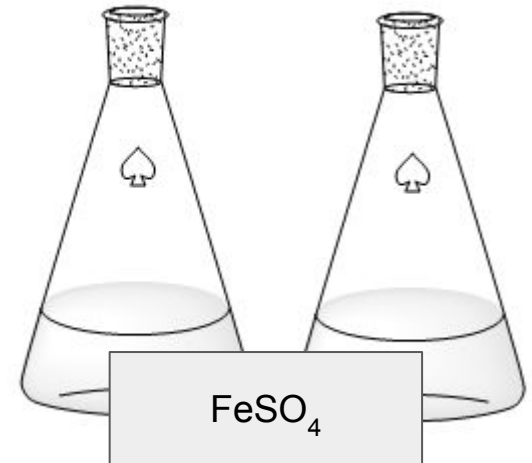


# Stepwise Procedure

4. Once reaction stops bubbling, transfer solutions into filter and perform vacuum filtration immediately.

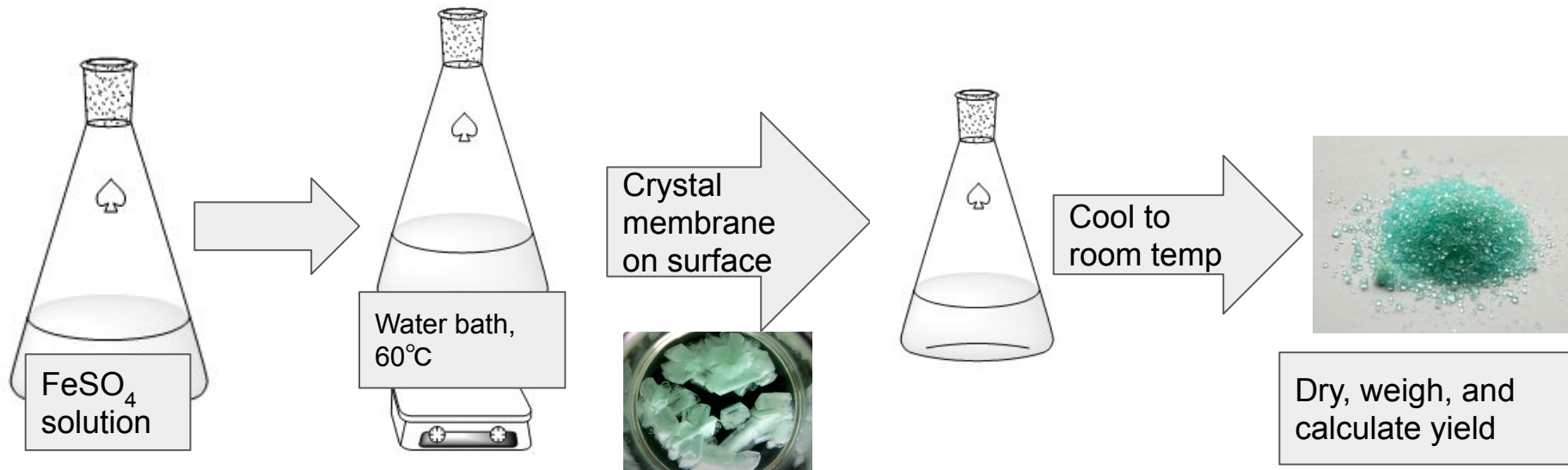


6. Disperse obtained solution into two small conical flasks.



# Stepwise Procedure

7. Heat one solution on the 60°C water bath until a crystal membrane is formed on the surface of the solution. Move solution out of water bath and allow to cool to room temp. Recover formed crystal, weigh, and calculate yield.



### 3. Preparation of $(\text{NH}_4)_2\text{SO}_4 \cdot \text{FeSO}_4$ Complex

1. Add saturated  $(\text{NH}_4)_2\text{SO}_4$  solution into another conical flask with  $\text{FeSO}_4$  solution.
2. Place solution in water bath (60-80 degrees C) for evaporation



$(\text{NH}_4)_2\text{SO}_4 : \text{Fe}$   
 $= 2.2/1.0$



$\text{FeSO}_4$



$\text{FeSO}_4$  flask  
sits in hot  
water bath

3. Remove container from water bath once crystal membrane has formed on the surface
4. Leave solution at room temperature for two hours
5. Recover  $(\text{NH}_4)_2\text{SO}_4 \cdot \text{FeSO}_4 \cdot 6 \text{H}_2\text{O}$  crystals and weigh

Removing  
 $\text{FeSO}_4$   
solution from  
hot water  
bath



(crystal membrane)

(leave  $\text{FeSO}_4$   
solution at room  
temperature for 2  
hours)



(recover crystals  
from solution)





#### 4. Measurement of trace amount of $\text{Fe}^{3+}$ in $(\text{NH}_4)_2\text{SO}_4 \cdot \text{FeSO}_4$ crystal

1. 1.0 g of  $(\text{NH}_4)\text{SO}_4 \cdot \text{FeSO}_4$  complex in 10 mL of oxygen free water



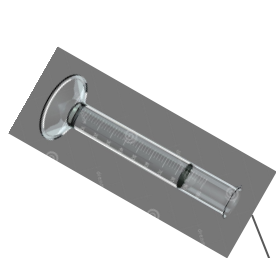
1.0 g of  
 $(\text{NH}_4)\text{SO}_4 \cdot \text{FeSO}_4$



10 mL water

2. Add 1.0 mL of 3.0 M  $\text{H}_2\text{SO}_4$  and 2.0 mL of 1.0 M KSCN to  $(\text{NH}_4)_2\text{SO}_4 \cdot 6 \text{H}_2\text{O}$  solution

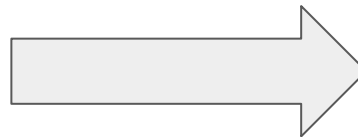
2.0 mL KSCN



1.0 mL  $\text{H}_2\text{SO}_4$



$(\text{NH}_4)_2\text{SO}_4 \cdot 6 \text{H}_2\text{O}$  solution



3. Transfer solution into a 25 mL volumetric flask, add more oxygen-free water to 25 mL



Fill to line



$\text{Fe}(\text{SCN})_n^{3-n}$  solution



4. Transfer 5.0 mL of the prepared solution into a 10 mL test tube, compare color to standard solution



Transfer 5.0 mL to test tube



(red indicates  $\text{SCN}^-$  has reacted with  $\text{Fe}^{3+}$ )



# Post Lab Questions

1. What instrument and technique can be used to give more accurate  $\text{Fe}^{3+}$  concentration in your prepared solution?

A spectrophotometer using spectrophotometry analysis technique could be used to give a more accurate  $\text{Fe}^{3+}$  reading. A linear calibration curve can be created with standard solutions and the concentration can be determined with a linear relationship to its absorption at a specific wavelength of light.

2. How could you reduce the  $\text{Fe}^{3+}$  contamination in your final products?

To reduce the  $\text{Fe}^{3+}$  contamination in your final products, it is important to use deoxygenated water and exposure to air because the  $\text{FeSO}_4$  reacts with oxygen and water to form the  $\text{Fe}(\text{OH})\text{SO}_4$  byproduct. It is also important to keep the solution warm to increase solubility of the  $\text{FeSO}_4$  and top up solvent to keep precipitate dissolved. It is also important to keep acid concentration high to prevent formation of OH.

