



## *Organic Chemistry Experiments*

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# **Preparation of Manganese(III) Acetylacetonate**



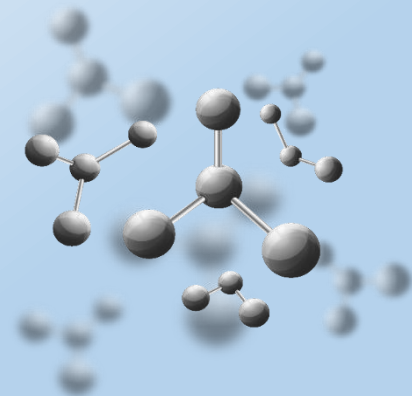


# Purposes and requirements

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**Complex** or **Coordination compound**, a class of substances in which a central metal atom is surrounded by other nonmetal atoms. Through the preparation of manganese(III) acetylacetonate, you can be familiar with the following knowledge:

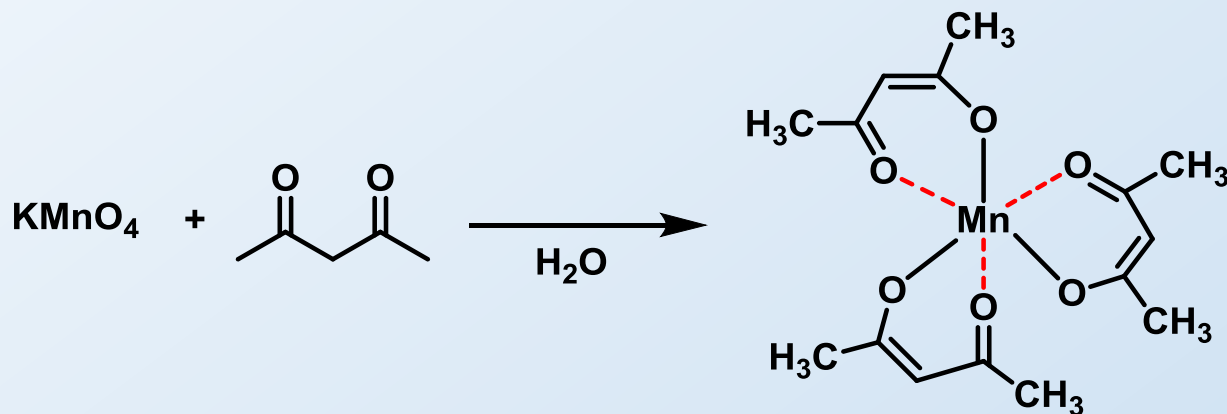
1. Master the synthetic methods of complexes
2. Master the operation of mixed solvent recrystallization
3. Review the manipulations of reflux and simple distillation reaction device



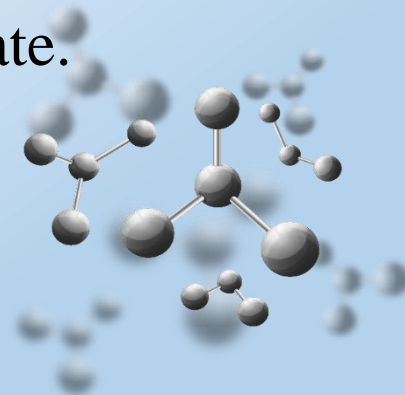


# Principle

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$\text{KMnO}_4$  undergoes a redox reaction with acetylacetone,  $\text{Mn}^{\text{VII}}$  is reduced to  $\text{Mn}^{\text{III}}$ , and then coordinated with acetylacetone to afford manganese(III) acetylacetonate.





## Reagents

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**Acetylacetone:**  $\text{CH}_3\text{COCH}_2\text{COCH}_3$   
b.p.  $139\text{ }^\circ\text{C}$

F.W. 100.11  
d. 0.976

**Potassium permanganate:**  $\text{KMnO}_4$

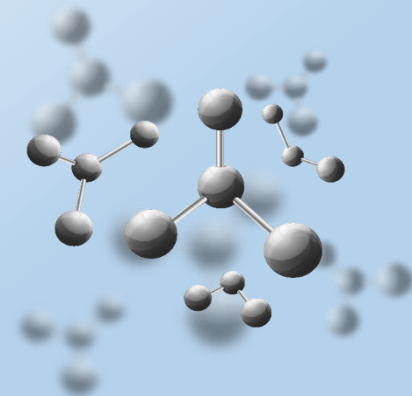
F.W. 159.00

**Acetone:**  $\text{CH}_3\text{COCH}_3$   
b.p.  $56\text{ }^\circ\text{C}$

F.W. 58.08  
d. 0.788

**Petroleum ether:** b.p.  $60\text{-}90\text{ }^\circ\text{C}$

**Manganese(III) acetylacetonate:**  $\text{Mn}(\text{C}_5\text{H}_7\text{O}_2)_3$   
b.p.  $60\text{-}90\text{ }^\circ\text{C}$

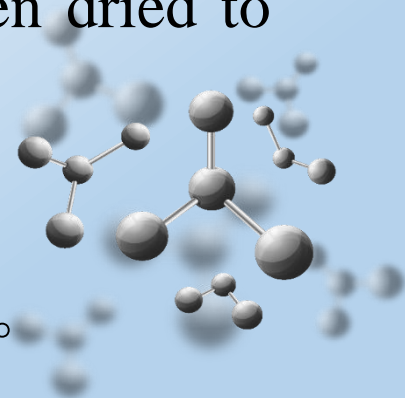




## Procedures

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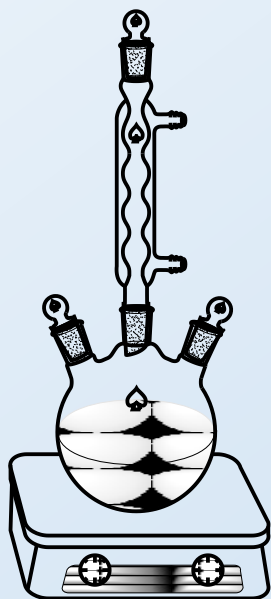
- 1)  $\text{KMnO}_4$  (1 g) +  $\text{H}_2\text{O}$  (10 mL), stirred at 75 °C;
- 2) Add acetylacetone 4.4 g (~ 4.8 mL) dropwise and stir at 75 °C for 10 min;
- 3) Remove heating, cooled with ice-water bath for 10 min ;
- 4) Filtered, washed with  $\text{H}_2\text{O}$  and 30% EtOH, then dried to constant weight;
- 5) Recrystallization with acetone-petroleum ether .



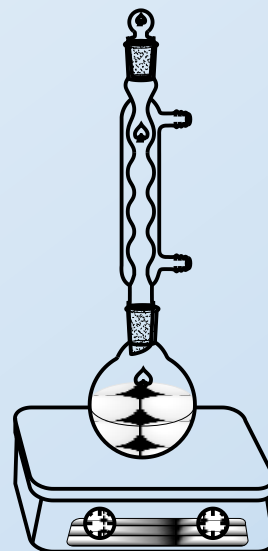


# Procedures

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◆ Apparatus for preparation



◆ Recrystallization



# Procedures

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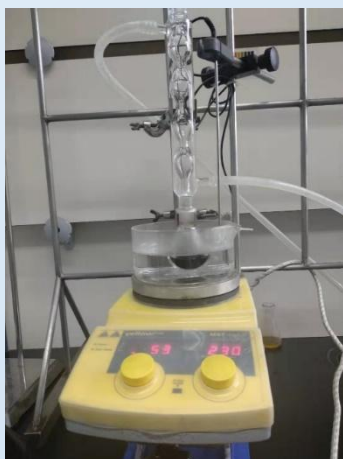
◆ preparation



◆ Filtered and washed



◆ Recrystallization



◆ Dried under IR







## Notes

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- 1) The reaction is very violent when initiating. Therefore, acetylacetone needs to be added dropwise.
- 2) The product has good solubility in ethanol. So the the concentration of EtOH should not be too high during washing.

