

Exp. 8: Oxidation: Preparation of Adipic Acid

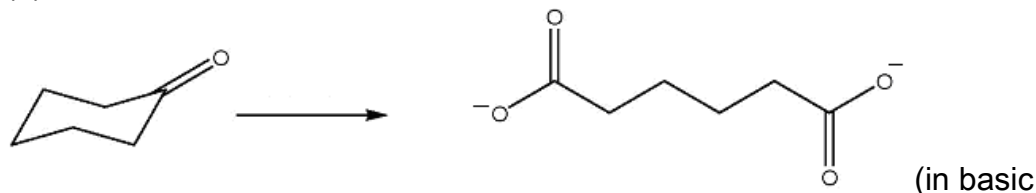
Pre-lab:

1. Balance the following main equation (in the introduction section) using the half-reaction method. (See your 112 notes or CHEM 112 text, pages 803-807.)

Show all your work at the beginning of the pre-lab write up. Whether you may get it right or wrong, you need to attempt and do until the last step.

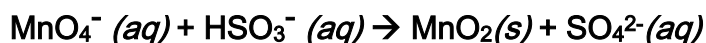
Hint:

(1) *The two half-reactions are:*



- (2) *assign oxidation numbers to the 2 C's that are oxidized;*
- (3) *add H₂O, H⁺, and e⁻ to balance O, H and the charge.*
- (4) *combine the two half-reactions by canceling the e⁻;*
- (5) *(for basic solution), add OH⁻ to neutralize H⁺ in the equation.*

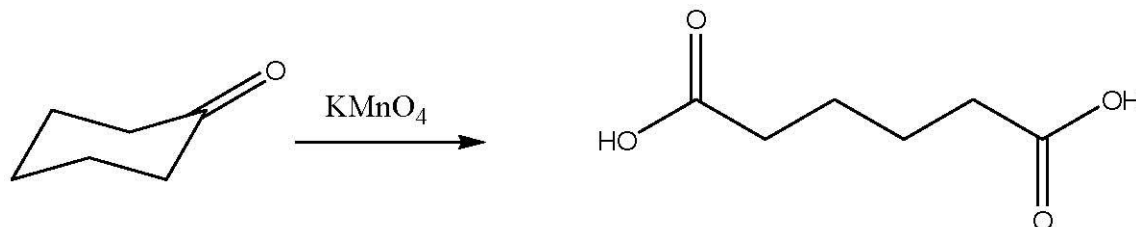
2. Balance the equation for step 4 using the half-reaction method: (you need to decide if the reaction is in acidic solution or basic)



3. Calculate the needed amount of cyclohexanone and of KMnO₄ in the first step of the procedure. Convert the units into mL for a liquid and grams for a solid.

Introduction:

Last week we saw the value of reduction reactions in chemistry, now we will see a simple oxidation reaction. Mild oxidizing agents do not react with ketones, but do react with aldehydes. In this case a very strong oxidizing agent is used to form a diacid, while breaking a carbon carbon bond.



Procedure:

1. In a 50 mL Erlenmeyer flask place 0.0025 moles of cyclohexanone and a solution of 0.0050 moles of potassium permanganate in 15 mL of water.
2. Make the solution slightly basic by adding 3 drops of 10% $\text{NaOH}(\text{aq})$.
3. Gently stir the solution for 10 minutes at room temperature, and then place it in a boiling water bath for 20 minutes. Once every 5 minutes, check to see if the oxidizing agent has been used up, by taking a drop of the reaction mixture and transferring it onto a filter paper. The appearance of a purple ring around the dark center is indicative of remaining potassium permanganate.
4. If after 20 minutes the oxidizing agent persists, it should be decomposed by addition of a small amount of sodium bisulfite.
5. Filter the solid and remove most of the manganese(IV) oxide. Wash the solids with 2 mL of hot water. Collect the water (filtrate).
6. Transfer the filtrate to a small beaker and concentrate the volume to less than 5 mL.

7. Add concentrated (12 M) HCl until acidic to litmus paper. Add another 10 drops of excess 12 M HCl. The product must have precipitated out at this point.
8. Cool the solution to room temperature, and collect the precipitated product by vacuum filtration.
9. Let the product air dry, and then measure its mass and the melting point.

For your report:

1. Calculate the % Yield of the adipic acid.
2. Predict the H-NMR spectrum of adipic acid.
3. Calculate the % error for the melting point from the literature value.