

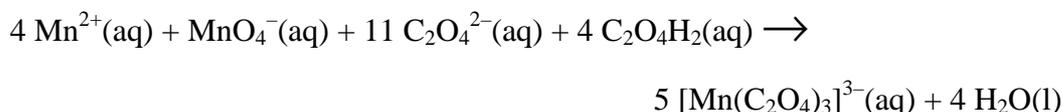
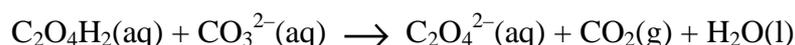
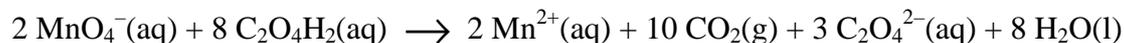
This experiment includes one preparation of a metal complex salt and two analyses of a provided sample of the same compound. The compound is a “classic” within inorganic photo chemistry.

Preparation of potassium tris(oxalato)manganate(III) hydrate, $K_3[Mn(C_2O_4)_3] \cdot xH_2O$

Note 1: The $[Mn(C_2O_4)_3]^{3-}$ ion is photosensitive and should therefore be protected from light as far as possible. Also, the thermal stability of the title compound is low.

Note 2: Before starting the synthesis, write down the thermometer reading in ice-water.

The synthesis comprises a reduction of manganese(VII) to manganese(II) with oxalic acid at 70 - 75 °C. After the addition of the sufficient amount of potassium ions in form of potassium carbonate, manganese(III) is formed by the addition of manganese(VII) at a temperature below 2 °C.



Dissolve 5.00 g of $\text{C}_2\text{O}_4\text{H}_2 \cdot 2\text{H}_2\text{O}$ in 35 mL of water in a 150 mL beaker by heating to 70 °C. Slowly add 1.00 g of KMnO_4 with magnetic stirring. The temperature must not exceed 70 - 75 °C. When the mixture is colourless, add 1.10 g of K_2CO_3 in small portions and cool the mixture in ice. When the temperature of the mixture has fallen to 25 - 30 °C, add 25 g of crushed ice. Meanwhile, cool the hotplate with a beaker containing ice. Maintain the temperature of the reaction mixture not more than 2 °C above your reported temperature of ice-water while adding 0.24 g of KMnO_4 in small portions with vigorous stirring. Stir for another 10 min and filter off the white precipitate and unmelted ice, if any, using the 60 mL filter syringe (See procedure A p. 13). Collect the filtrate in a 250 mL beaker cooled in ice. Add 35 mL of ice-cold ethanol to the cherry-red filtrate (just swirl the beaker; stirring will lead to the formation of tiny crystals), wrap the beaker in aluminium foil and cool it in ice for 2 h (swirl the beaker three or four times during this period).

Clean the filter - first with 4 M HCl, then with water. Collect the cherry-red crystals by filtration using the 60 mL filter syringe, wash first with 2×5 mL of ethanol and then with 2×5 mL of acetone, and dry the product in air and protect it from light for at least one hour. A brown vial with lid should be taken to be tared by the lab. assistant. When dry, the product is placed in the vial. Write name and student code on

the vial. Then close the vial and take it and your answer sheet to the lab. assistant who will weigh your sample. The theoretical yield is 7.6 mmol.

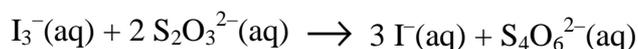
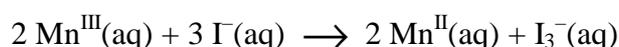
1-A Record the yield in grams.

1-B Suggest a molecular formula of the white precipitate which is removed in the first filtration.

Analysis of the provided sample of $\text{K}_3[\text{Mn}(\text{C}_2\text{O}_4)_3] \cdot x\text{H}_2\text{O}$ for oxidizing ability

Note 3: The burette contains a cleanser and should therefore be rinsed 3 - 4 times with water before use.

Manganese(III) is reduced to manganese(II) by iodide ions and the triiodide ions formed are then titrated with thiosulfate.



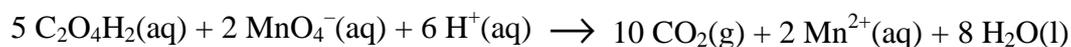
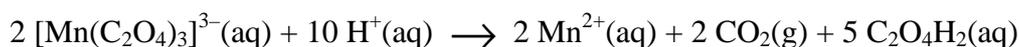
In a 250 mL conical flask dissolve 1.0 g of KI in 25 mL of demineralized water and add 10 mL of 4 M HCl. Immediately after an accurately preweighed sample (approx. 200 mg) of the provided complex is transferred (as much as possible is poured directly into the liquid in small portions before the residue is washed down) quantitatively with demineralized water to the flask. Titrate the I_3^- formed with the standardized, approx. 0.025 M $\text{Na}_2\text{S}_2\text{O}_3$ solution. When the brown colour has faded to light yellow, add 2 mL of starch indicator solution and continue the titration until the colour changes from blue to colourless.

1-C Calculate the molar mass of the analyzed compound from the titration data.

Analysis of the provided sample of $\text{K}_3[\text{Mn}(\text{C}_2\text{O}_4)_3] \cdot x\text{H}_2\text{O}$ for reducing ability

Note 4: The burette should be rinsed 2 - 3 times with water before this titration.

Manganese(III) is reduced to manganese(II) by the oxalate ligands, and excess oxalate is titrated with permanganate.



Transfer an accurately preweighed sample (approx. 200 mg) of the provided complex quantitatively with demineralized water to a 250 mL conical flask. Add 25 mL of 2 M sulfuric acid and heat the solution to 75 - 80 °C. Without further heating, titrate with the standardized, approx. 0.025 M KMnO_4 solution. Near the end of the titration add the titrant slowly, until one drop gives the solution a rose colour which does not fade on standing for 0.5 min.

1-D Calculate the molar mass of the analyzed compound from the titration data.

The results of the two types of analysis may differ by up to 10 %. Use only the result from the titration with KMnO_4 for the following calculation.

1-E Calculate the value of x in the formula $\text{K}_3[\text{Mn}(\text{C}_2\text{O}_4)_3] \cdot x\text{H}_2\text{O}$ and the yield of your preparation in percent of the theoretical yield.