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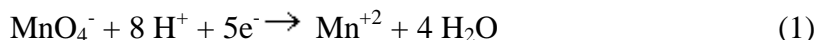
## Redox Titration – Potassium Permanganate with an Iron(II) Salt

### Objective:

To use a standardized potassium permanganate solution to determine the percentage of iron in an unknown sample.

### Abstract:

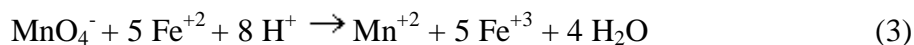
Oxidation numbers describe the number of electrons the atom will gain or lose during a reaction. Each atom in an equation can be assigned an oxidation number according to certain rules. If the oxidation number of an atom increases as you go from the left side to the right side of an equation, oxidation has occurred (electrons have been lost); if the oxidation number decreases, reduction has occurred (electrons have been gained).



In the above reduction half-reaction, manganese has undergone a decrease in oxidation number from +7 to +2. In the following oxidation half-reaction, each iron atom has undergone an increase in oxidation number from +2 to +3.



Oxidation must occur along with reduction. These reactions are called redox (reduction/oxidation) reactions. The number of electrons lost and gained in the half-reactions must be equal. The overall redox reaction becomes:

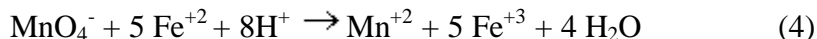


In Part A of the experiment you will standardize a  $\text{KMnO}_4$  solution by titrating it against a ferrous ammonium sulfate ( $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ ) solution where the number of moles of ferrous ammonium sulfate is precisely known. Using a buret you will slowly add the  $\text{KMnO}_4$  solution to the ferrous ammonium sulfate solution as shown in Equation 3.  $\text{KMnO}_4$  acts as its own indicator. That is, at the start of the titration, the deep violet color of  $\text{MnO}_4^-$  will be lost because it is changing to  $\text{Mn}^{+2}$ , but as soon as there is no more  $\text{Fe}^{2+}$  in the solution for the  $\text{MnO}_4^-$  to react with, the  $\text{MnO}_4^-$  will remain in the reaction solution. The pink color of the dilute  $\text{MnO}_4^-$  solution indicates the end of the reaction. This is known as the endpoint or equivalence point of the titration.

Knowing the mass of ferrous ammonium sulfate to the nearest 0.01g, you can precisely determine the number of moles of ferrous ammonium sulfate. This will be equal to the number of moles of  $\text{Fe}^{2+}$  ions in solution. Using equation (3), one can then determine the number of moles of  $\text{MnO}_4^-$  that have reacted during the titration. Knowing the milliliters of  $\text{MnO}_4^-$  solution used in the titration, the molarity of the  $\text{MnO}_4^-$  can be calculated

because molarity is equal to the moles of solute ( $\text{MnO}_4^-$ ) per liter of solution. This will be equal to the molarity of the  $\text{KMnO}_4$  solution as one mole of  $\text{KMnO}_4$  dissolves in water to give one mole of  $\text{MnO}_4^-$ . That is another way of saying that you have standardized the  $\text{KMnO}_4$  solution.

Part B of the experiment is very similar. The standardized  $\text{KMnO}_4$  solution is used to determine the percentage by mass of Fe in a sample. The concentration of the  $\text{KMnO}_4$ , remains the same as Part A, but the amount of  $\text{Fe}^{+2}$  present in the sample is unknown. The titration is performed in the same way.



Since we know the precise molarity of the  $\text{MnO}_4^-$  solution from Part A, multiplying liters of  $\text{MnO}_4^-$  solution used in a titration times moles of  $\text{MnO}_4^-$  per liter of solution (its molarity) the moles of  $\text{MnO}_4^-$  that have reacted is obtained. Using reaction 3 the number of moles of  $\text{Fe}^{+2}$  is determined. This will allow us to calculate the number of grams of Fe in the sample. Finally, knowing the mass of the sample, the percent iron in the sample used in the titration is calculated.

Equipment:

250 mL Erlenmeyer flask	buret
ring stand	buret clamps
balance	100 mL graduated cylinder

Materials:

$\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$	0.02 M $\text{KMnO}_4$
3 M $\text{H}_2\text{SO}_4$	unknown $\text{Fe}^{+2}$ sample
concentrated $\text{H}_3\text{PO}_4$	

Procedure:

Part A – Standardization of the Potassium Permanganate Solution

1. Rinse a buret thoroughly with distilled water. Rinse the buret carefully once with a 5 mL portion of  $\text{KMnO}_4$  solution. After these rinses, fill the buret with the  $\text{KMnO}_4$  solution. Be sure that the tip of the stopcock is filled.
2. Mass a 0.5 g sample of dried ferrous ammonium sulfate,  $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6 \text{H}_2\text{O}$ , on a balance to the nearest 0.01 g. Record the mass in your lab notebook. Place the sample in a 250-mL Erlenmeyer flask. Dissolve the sample in 75 mL of distilled water. Add 5 mL of 3 M  $\text{H}_2\text{SO}_4$ , and 3 mL concentrated  $\text{H}_3\text{PO}_4$  (85%) to the flask.

Add a magnetic stir bar to the flask. The  $\text{Fe}^{+3}$  that will be produced during the titration forms a colorless complex with the  $\text{PO}_4^{-3}$  ions. This simplifies the detection of the endpoint.

3. Record the initial reading on the buret (read the bottom of the meniscus at eye-level) to the nearest 0.01 mL. Start to add the  $\text{KMnO}_4$  solution. When the violet color of the  $\text{MnO}_4^-$  ion in the reaction does not disappear quickly, add the solution slowly. Towards the end of the titration, the solution should be added one drop at a time. When a faint pink color persists for 30 seconds, with constant swirling, the end-point has been reached. A white piece of paper under the Erlenmeyer flask will aid in detecting color changes.
4. Record the level of  $\text{KMnO}_4$  solution in the buret to 0.01 mL.
5. Write your mass and volume used in the chart on the chalkboard. The entire class's data will be used to find the molarity.

#### Part B

1. Mass a 0.4 g sample of the unknown mixture containing  $\text{Fe}^{+2}$  ion a balance to the nearest 0.01 g. Be sure to record the unknown letter. Place the sample in a 250 mL Erlenmeyer flask. Dissolve the sample in 75 mL of distilled water. Add 5 mL of 3 M  $\text{H}_2\text{SO}_4$ , and 3 mL concentrated  $\text{H}_3\text{PO}_4$  (85%) to each flask.
2. Refill the buret to the 0 mL mark. Titrate the unknown solution with the standardized  $\text{KMnO}_4$  solution. The procedure is the same as in the above titrations. Again, towards the end of the titration, the  $\text{KMnO}_4$  solution has to added one drop at a time.
3. Empty unused  $\text{KMnO}_4$  solution into the appropriate bottle. Rinse your buret several times with water. All contents of the reaction flask may be disposed of into the sink.

#### Calculations:

1. Use the class's data from the standardization to find the molarity of potassium permanganate solution. Be sure to perform a Q-Test on all data pieces before averaging them.
2. Determine the number of moles of  $\text{Fe}^{2+}$  reacted in each Part B trial using the  $\text{KMnO}_4$  molarity and reaction 4. Convert these values to grams and determine the percentage of iron in the sample. Report an average percentage after performing a Q-Test.