EXPERIMENT 2: Determination of Oxalate in Potassium Trisoxalatoferrate(III)

Introduction

In this experiment, you will use a standard solution of potassium permanganate to analyze the oxalate content of the potassium trisoxalatoferrate(III) trihydrate, K₃[Fe(C₂O₄)₃]·3H₂O, that you prepared in last week’s procedure in order to determine its purity.

Potassium permanganate is a useful REDOX titrant because it is its own indicator. The intense purple colour of the permanganate ion changes to the very pale pink (almost colourless) of manganese(II) ion as the permanganate ion is reduced. The very intensely red colour of the permanganate solution makes the bottom of the meniscus in the burette very difficult to see and, in this case only, the TOP of the meniscus may read from the burette.

Permanganate ion, MnO₄⁻, can be reduced in a variety of ways depending on the reaction conditions, especially pH. In neutral solution one of the main products is hydrated manganese(IV) oxide. This at first gives the solution a brown colour and finally gives a brown precipitate. Under alkaline conditions the permanganate ion is reduced to the green manganate ion, MnO₄²⁻. Under acid conditions the permanganate ion is reduced further to the very pale pink (almost colourless) manganese(II) ion. For this titration, reduction to the manganese(II) ion is required, so the titration must be done under acidic conditions.

Permanganate ion oxidizes oxalate ion to carbon dioxide gas in the following reaction.

\[ 2 \text{MnO}_4^- (aq) + 16 \text{H}^+ + 5 \text{C}_2\text{O}_4^{2-} \rightarrow 2 \text{Mn}^{2+}(aq) + 10 \text{CO}_2(g) + 8 \text{H}_2\text{O(l)} \]

In acid solution, potassium trisoxalatoferrate(III) provides Iron(III) and oxalate ions. Iron(III) is not oxidized by permanganate ion so the oxalate can be titrated directly.

However, oxalate ions react only slowly with permanganate ions at room temperature so the solution must be warmed to about 60 °C in order for the reaction to be fast enough to be useful in a titration.

Objectives
1. to practice skills required for quantitative volumetric analysis.
2. to learn how titrations are used to monitor the quality of industrial products.
3. to gain further experience with stoichiometric calculations and the use of the mole.
Procedure

1. Collect about 200 mL of the standard potassium permanganate solution in a small clean dry beaker and cover with a watch glass. Label it with the concentration (mol L⁻¹) as written on the stock bottle. Note this concentration in your report.

2. Obtain a 50 mL burette and rinse carefully 3 times with tap water followed by 3 times with deionized water.

3. Similarly, rinse the burette three times with a little of the potassium permanganate solution, then fill it with the solution until it is just above the 0.00 mark. Run a little of the solution out of the burette to allow any air bubbles to escape from the tip. There is no need to adjust the burette to 0.00 mL so long as the meniscus is on the scale.

4. Weigh approximately 0.2 - 0.25 g of your potassium trisoxalatoferrate(III) trihydrate product into a clean dry weighing vial using the top loading balance and then weigh the vial and contents on the analytical balance. Prepare a clean 250 mL conical flask and, using a clean funnel, transfer the solid to the flask. Reweigh the empty vial on the analytical balance. Record the mass of the sample used in the table.

5. Add 25 mL of 3.0 mol L⁻¹ sulfuric acid to the solid rinsing the last traces of solid in the funnel into the flask. Swirl the mixture until the solid has dissolved completely. Warm the solution in the flask until it is steaming hot on the hot plate provided.

6. Record the initial burette reading to 2 decimal places. If the solution is too deeply colored it is acceptable to read the top of the meniscus.

7. Now titrate the hot solution with the potassium permanganate solution reheating to 60°C near the endpoint if necessary. Swirl the flask continuously while permanganate is added until the first pale yellowish-pink colour appears. If the colour fades, add more permanganate carefully. Record the final reading from the burette to 2 decimal places. Discard the contents of the flask. Rinse it once with tap water and once with deionized water.

8. Repeat the titrations at least twice with additional 0.2 g samples of the potassium trisoxalatoferrate(III) until consistent results are obtained. The ratio of the volume relative to the sample mass should be within 0.5% of each other.

9. RECORD ALL OF YOUR RESULTS DIRECTLY INTO YOUR LABORATORY REPORT.
Data Table

Format for Recording Titration Results

Titration of Potassium Trisoxalatoferrate(III) with Standard Potassium Permanganate

Concentration of potassium permanganate in burette: ________________________ mol L\(^{-1}\)

Color change at end point _________________________

<table>
<thead>
<tr>
<th></th>
<th>Lowest mass</th>
<th>→</th>
<th>Highest mass</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Trial 1</td>
<td>Trial 2</td>
<td>Trial 3</td>
</tr>
<tr>
<td>Mass of vial and contents</td>
<td>g</td>
<td>g</td>
<td>g</td>
</tr>
<tr>
<td>Mass of emptied vial</td>
<td>g</td>
<td>g</td>
<td>g</td>
</tr>
<tr>
<td>Mass of trisoxalatoferrate(III) trihydrate</td>
<td>g</td>
<td>g</td>
<td>g</td>
</tr>
<tr>
<td>Burette Readings</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Final</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Initial</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Volume used (mL)</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

\[
\frac{volume (mL)}{mass (g)}
\]

Volume/mass ratios must be within 0.5% of each other
Calculations

1. For the two best trials (within 0.5% of each other), calculate the number of moles of KMnO₄ that was present in the solution delivered by the burette.

2. For the two best trials, calculate the number of moles of oxalate, C₂O₄²⁻, that reacted with the KMnO₄(aq) based on the reaction stoichiometry.

3. Calculate the mass in grams of oxalate, C₂O₄²⁻, in each of the samples of K₃[Fe(C₂O₄)₃]·3H₂O, titrated by standard KMnO₄(aq).
4. Use the formula for potassium trisoxalatoferrate(III) trihydrate, 

$$\text{K}_3[\text{Fe}(\text{C}_2\text{O}_4)_3] \cdot 3\text{H}_2\text{O}$$

to calculate the theoretical percentage by mass of oxalate, $\text{C}_2\text{O}_4^{2-}$, in the compound.

5. Based on your answers to question 3, calculate the percentage by mass of oxalate, $\text{C}_2\text{O}_4^{2-}$, in each of the samples of $\text{K}_3[\text{Fe}(\text{C}_2\text{O}_4)_3] \cdot 3\text{H}_2\text{O}$. Average the two answers.

6. Calculate the % purity: 

$$\frac{\text{observed } \% \text{ C}_2\text{O}_4^{2-}}{\text{theoretical } \% \text{ C}_2\text{O}_4^{2-}} \times 100$$
Determination of Oxalate in Potassium Trisoxalatoferrate(III).

The following experimental data were obtained by a typical student:

Titration of Potassium Trisoxalatoferrate(III) with standard potassium permanganate

Concentration of KMnO$_4$(aq) in burette: 0.02058 mol L$^{-1}$

Solid used: $K_3[Fe(C_2O_4)_3]·3H_2O$

<table>
<thead>
<tr>
<th>Trial 1</th>
<th>Trial 2</th>
<th>Trial 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mass of solid (g)</td>
<td>0.1882</td>
<td>0.2198</td>
</tr>
</tbody>
</table>

Burette readings

<table>
<thead>
<tr>
<th>Trial 1</th>
<th>Trial 2</th>
<th>Trial 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Final volume (mL)</td>
<td>26.08</td>
<td>29.79</td>
</tr>
<tr>
<td>Initial volume (mL)</td>
<td>3.09</td>
<td>3.15</td>
</tr>
<tr>
<td>Volume used (mL)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Vol/mass ratio</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

1. For the two best trials (within 0.5% of each other), calculate the number of moles of KMnO$_4$ that was present in the solution delivered by the burette.

2. For the two best trials, calculate the number of moles of oxalate, C$_2$O$_4^{2-}$, that reacted with the KMnO$_4$(aq) based on the reaction stoichiometry.
3. Calculate the mass in grams of oxalate, $C_2O_4^{2-}$, in each of the samples of $K_3[Fe(C_2O_4)_3] \cdot 3H_2O$, titrated by standard $KMnO_4(aq)$.

4. Use the formula for potassium trisoxalatoferrate(III) trihydrate,

$$K_3[Fe(C_2O_4)_3] \cdot 3H_2O$$

to calculate the theoretical percentage by mass of oxalate, $C_2O_4^{2-}$, in the compound.

5. Based on your answers to question 3, calculate the percentage by mass of oxalate, $C_2O_4^{2-}$, in each of the samples of $K_3[Fe(C_2O_4)_3] \cdot 3H_2O$. Average the two answers.

6. Calculate the percentage purity of the product.

$$\frac{\text{observed } \% \ C_2O_4^{2-}}{\text{theoretical } \% \ C_2O_4^{2-}} \times 100\%$$