

Skills Challenges – CH1020

Determination of Unknowns – Post Lab Submission

Daniel McDonough, Muntasir Shahabuddin, Jonathan Scribner

Description of unknown: ____ Concentration of NaOH (aq) and H₂SO₄ (aq)_____

Equipment/device(s) used:

Burette, beaker

Reason for using each measuring device:

Beaker holds unknown and burette measures amount of titrant required to neutralize analyte

Limitations of each measuring device used:

Burette graduation limits accuracy, and going past the equivalence point creates a discrepancy between the true and calculated concentration of the analyte.

Procedure (*Be concise and comprehensive, use grammatically correct complete sentences*):

1. Standardize NaOH Solution
 - a. Mass a cleaned 250 mL beaker
 - b. Add about .5 grams of KPH and mass the beaker after adding KPH to obtain exact mass of KPH
 - c. Add exactly 35 mL of distilled water to 250 mL beaker and dissolve KPH. Calculate molarity of KPH
 - d. Fill burette with KPH solution
 - e. Fill 150 mL beaker with 5 mL of unknown NaOH solution
 - f. Add 3 drops of PTH to NaOH in 150 mL beaker
 - g. Titrate KPH solution into unknown concentration of NaOH. Titrate until NaOH and PTH solution in 150 mL beaker is clear and colorless
 - h. Record final volume of KPH in burette and calculate molarity of NaOH based on moles and volume of KPH consumed
2. Fill burette with NaOH solution and record initial volume
3. In 250mL beaker, add 5 mL of unknown concentration of H₂SO₄
4. Add 3 drops of PTH indicator to H₂SO₄ beaker
5. Slowly drip known concentration of NaOH into unknown concentration of H₂SO₄ while stirring contents of 250mL beaker until indicator becomes a very light pink
6. Record final volume of NaOH in burette

Results

a. Table(s) Showing All Data & Calculations (refer to Conference Worksheets)

	Trial 1	Trial 2	Trial 3
Moles KHP	.00280	.00280	.00280
Moles NaOH	.00133	7.52×10^{-4}	6.97×10^{-4}
[NaOH] in mol/L	.133	.151	.140
%RSD	6.42%		

	Trial 1	Trial 2	Trial 3
Moles NaOH	.00110	.00110	.0110
Moles H₂SO₄	.00133	.00128	.00130
[H₂SO₄] in mol/L	.266	.255	.260
%RSD	2.12%		

b. Show Complete Calculation for Determining Unknown Concentration for 1 run

$$.5625\text{g}/204.22\text{g/mol} = .0028 \text{ mol KHP}$$

$$.0028\text{mol}/.035\text{L} = .0787 \text{ M KHP}$$

$$V_{\text{KHP}} = 9.6 \text{ mL of } .0787 \text{ M KHP}$$

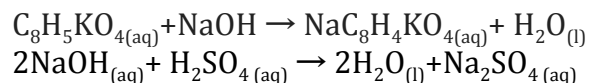
$$\frac{(.0096\text{L})(.00787\text{M})}{.005 \text{ L NaOH}} = .151\text{M NaOH}$$

.141 M NaOH from standardization

$$V_{\text{NaOH}} = .0189 \text{ L NaOH}$$

$$(.0189\text{L NaOH}) * (.141\text{M NaOH}) * (1\text{mol H}_2\text{SO}_4 / 2\text{mol NaOH}) * 1 / .005\text{L H}_2\text{SO}_4 = .266 \text{ M H}_2\text{SO}_4$$

c. Write Balanced Equation:



Discussion

Suggestions for All: Discuss what sources of error are present in your experiment. Discuss how your calculated unknown concentration compares to the suggested unknown concentration? What are the reasons for differences?

During the titration of the NaOH, improper creation and measurement of the mass and concentration of the KHP would cause the standardization to become inaccurate. This causes error in the calculation of the concentration of NaOH. Since the NaOH solution is then used to determine the concentration of the H_2SO_4 , error in the standardization would translate to error in the calculation of the H_2SO_4 solution concentration. While the NaOH can be re-standardized and the new standardization used to recalculate the concentration of the H_2SO_4 , errors in the standardization will stack into errors in the real concentration of the H_2SO_4 .

Description of unknown: _____ Concentration of Fe^{2+} _____

Equipment/device(s) used:

Burette, beaker

Reason for using each measuring device:

Beaker holds unknown and burette measures amount of titrant required to neutralize analyte

Limitations of each measuring device used:

Burette graduation limits accuracy, and going past the equivalence point creates a discrepancy between the true and calculated concentration of the analyte.

Procedure (*Be concise and comprehensive, use grammatically correct complete sentences*):

1. Wash burette with KMnO_4 solution
2. Fill burette with KMnO_4 solution and record initial volume
3. Wash 250 mL beaker with water
4. Add 5 mL of unknown Fe^{2+} solution to beaker
5. Titrate KMnO_4 solution into the 250 mL beaker until the magenta color in the beaker disappears.
6. Record final volume of KMnO_4 solution in burette

Results

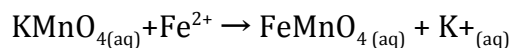
a. Table(s) Showing All Data & Calculations (refer to Conference Worksheets)

	Trial 1	Trial 2	Trial 3
Moles KMnO_4	1.00×10^{-4}	1.00×10^{-4}	9.80×10^{-5}
Moles Fe^{2+}	2.00×10^{-5}	2.00×10^{-5}	1.96×10^{-5}
$[\text{Fe}^{2+}]$ in mol/L	.00400	.00400	.00392
%RSD	1.16%		

b. Show Complete Calculation for Determining Unknown Concentration for 1 run

$$\frac{.01 \text{ L MnO}_4^-}{1} * \frac{.01 \text{ mol MnO}_4^-}{1 \text{ L MnO}_4^-} * \frac{1 \text{ mol Fe}^{2+}}{5 \text{ mol MnO}_4^-} * \frac{1}{.005 \text{ L Fe}^{2+}} = .00400 \text{ M Fe}^{2+}$$

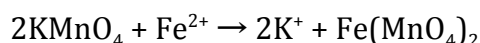
c. Write Balanced Equation:



Discussion

During the redox titration, the KMnO_4 did not properly change colors to the slightly green equivalence point marker. The titration skipped the green hue and immediately turned very slightly pink during all trials, with no transition to green at all during any of the trials.

The reaction between the Fe^{2+} and the KMnO_4 proceeds as shown:



The indication of the titration endpoint takes advantage of KMnO_4 's vibrant magenta color. As the KMnO_4 reacts with the Fe^{2+} ions, the vibrant color is replaced by a colorless solution as $\text{Fe}(\text{MnO}_4)_2$ reacts to replace the colored KMnO_4 . The equivalence point is indicated by a greenish hue due to the color of Fe^{2+} in that specific oxidation state being equal in concentration to the MnO_4^- . The error is likely due to overshooting the equivalence point and leaving too much KMnO_4 left unreacted. The light pink color indicates that our titrations were barely over the equivalence point. Since this occurred for every trial, it is likely that our overall calculation for the concentration of Fe^{2+} will be higher than the true value (due to a higher measured usage of KMnO_4).

Description of unknown: _____ Concentration of Cu^{2+} _____

Equipment/device(s) used:

Erlenmeyer flask, filter paper, funnel, scale

Reason for using each measuring device:

Filter paper separates precipitate from the solution, and the scale masses the amount of precipitate formed

Limitations of each measuring device used:

The scale can only accurately display a mass to a certain amount of significance, and there could be imprecisions creating systematic error in the scale.

Procedure (*Be concise and comprehensive, use grammatically correct complete sentences*):

1. Mass a 250 mL beaker
2. Wash burette with known concentration of Na_2CO_3
3. Fill burette with Na_2CO_3 and measure initial volume in burette
4. Wash 250 mL beaker and add 5 mL of unknown concentration of CuSO_4 into beaker
5. Titrate Na_2CO_3 solution into 250 mL beaker while slowly swirling contents of 250mL beaker until no more precipitate forms
6. Allow precipitate to settle
7. Decant as much liquid as possible without removing any precipitate
8. Place beaker on hot plate and evaporate liquid until just precipitate remains
9. Mass beaker with only precipitate in it. Use initial mass to calculate mass of precipitate

Results

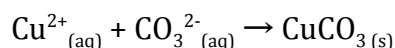
a. Table(s) Showing All Data & Calculations (refer to Conference Worksheets)

	Trial 1	Trial 2	Trial 3
Moles CuSO_4	.00597	.00665	.00626
$[\text{CuSO}_4]$ in mol/L	1.19	1.33	1.25
%RSD	5.59%		

b. Show Complete Calculation for Determining Unknown Concentration for 1 run

$$\begin{aligned} (.738 \text{ g CuCO}_3)/123.55\text{g/mol} &= .00597 \text{ mol Cu}^{2+} \\ .00597\text{mol}/.005\text{L} &= 1.19 \text{ M Cu}^{2+} \end{aligned}$$

c. Write Balanced Equation:



Discussion

There was a series of possible sources of error during the precipitation titration as well. There were very small amounts of precipitate that we could not wash out of the flask after the reaction occurred. Any precipitate left behind in the reaction vessel will lower the calculated concentration of Cu^{2+} ions due to a lower measured mass of copper precipitate. Since titration was not required as long as we collected the mass of the precipitate, we added excess Na_2CO_3 . However, after the precipitation reaction, the liquid that was filtered from the precipitate was still blue, indicating an incomplete precipitation (and thus additional copper (ii) ions left in solution. In order to fully precipitate all copper (ii) ions in the solution, we added additional Na_2CO_3 . However, no additional precipitation occurred and the solution remained blue. Since a portion of the copper (ii) ions were left in solution, our calculation for the concentration of copper (ii) ions in the original solution will be lower than the true value. It is possible that the oxidation state of copper in the given solution of CuSO_4 was not purely Cu^{2+} and could have been Cu^+ which would change its reactivity in relation to its higher oxidation state counterpart. We also observed that the color of the solution that was separated from the precipitate was a darker blue than the

original CuSO_4 solution, further indicating the presence of a different salt, whether a result of the valency difference in the copper ions, or something else altogether.

Further discussion:

The solutions that will remain the most stable in solution are the strong acid and base, H_2SO_4 and NaOH respectively. Because sodium and hydrogen only have one realistic oxidation state when in these solutions, they are likely to stay in said state and keep the solution stable in its composition as long as water does not evaporate off. However, the two metal salts, containing iron and copper, have two possible oxidation states each. Between the two metal salts, Fe^{2+} is less stable as an ion, as seen in the formation of iron oxides versus copper oxides. Copper, when exposed to oxygen, takes significantly longer to oxidize than iron does. This is confirmed by iron's higher place in the metal activity series relative to copper. Considering this, it is likely for iron to change valency into Fe^{3+} , changing the concentration of Fe^{2+} ions in solution. Although, similar logic can be applied to Cu^{2+} changing oxidation states to Cu^+ . This change in oxidation states in either metal ion could explain a degree of error in our data--especially in the precipitation reactions where copper salts were still present even after adding excess CO_3^{2-} , as discussed above.