CHEM 1002A

Reduction-Oxidation Titration

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Date Performed: 05/02/2019
Date Submitted: 26/02/2019

Purpose:

The purpose of this lab is to use reduction-oxidation titration to determine the unknown percent quantity of the element Iron (Fe^{2+}) in a sample using a standardized potassium Permanganate solution.

Theory:

Titration using redox reaction can be based similarly to acid-base reactions for many reasons. Firstly, the reaction must be thermodynamically spontaneous enough to be stoichiometric. Secondly, the reaction must be kinetically fast enough to give instant results. Lastly, during the process, it is impossible for side reactions to occur and an indicator must present (Robert Burk Chem1002). There are a variety of steps in order to analyze Fe^{2+} by redox titration. In this experiment one of the oxidation reagents used to carry out this fete is potassium permanganate.

Redox titration of Fe^{2+} *with* $KMnO_4$ *:*

Half reactions:

Oxidation of Fe^{2+} ; $Fe^{2+} \rightarrow Fe^{3+} + 1\ e^-$

Reduction of MnO_{4-} ; $MnO_{4-} + 8H3O^+ \rightarrow Mn^{2+} + 12\,H_2O$

In the final stoichiometric equation, the K+ is often removed as it is a spectator ion

$$MnO_4^-{}_{(aq)} + 8H_3O^+{}_{(aq)} + 5Fe^{2+}{}_{(aq)} \rightarrow 5Fe^{3+}{}_{(aq)} + Mn^{2+}{}_{(aq)} + 12H_2O_{(l)}$$

- In order to speed up the process of equilibrium, add sulfuric acid(H_2SO_4), and have the titration going at high temperatures because the sulfuric acid acts as a catalyst allowing the equilibrium to shift to the right.

- During this process of titration, Mn O_{4-} is reacting with Mn^{2+}, this means that when the acid solution gradually fades into a more purplish colour the end point can be observed in split second.
- The purpose for adding phosphoric acid is mainly because of its colour formation because the hydrated Fe^{3+} is orange and it can easily distribute the end point of the reaction. When you add the phosphoric acid, the solution becomes colourless.

$$2MnO_4^-{}_{(aq)} + 3Mn^{2+}{}_{(aq)} + 6H_2O_{(l)} \rightarrow 5MnO_{2(s)} + 4H_3O^+{}_{(aq)}$$

The best way to read a buret is to shine a light directly behind your viewpoint of the buret.

- In this experiment, side reactions should mainly be avoided. You know you have inherited a side reaction when a brown precipitate is formed which is when the reaction produces MnO_2 resulting in a neutral solution. Also any Cl ions should be avoided in the flask as they could possibly mix with Mn O_{4-} and react with oxidizing Cl anions.

Standardization of $KMnO_4$ solution with $Na_2C_2O_4$:

- Standardization reaction:

Oxidation of $C_2O_4^{2-}$: $C_2O_4^{2-} \rightarrow 2CO_2 + 2e^-$

Reduction of MnO_4^-: $MnO_4^- + 8H_3O^+ + 5e^- \rightarrow Mn^{2+} + 12H_2O$

The overall stoichiometric reaction requires 5 times of the oxidation as reduction runs twice

$$16\ H_3O^+{}_{(aq)} + 5C_2O_4^{2-}{}_{(aq)} + 2MnO_4^-{}_{(aq)} \rightarrow 2Mn^{2+}{}_{(aq)} + 10CO_{2(g)} + 24H_2O_{(l)}$$

- $KMnO_4$ is unstable in air, the reason being is because with the presence of light, MnO_2 could form when it comes into contact with certain contaminants in a solution. Alkaline solutions are less stable than neutral, so any presence of light can cause it to decompose.

- The reason why the $KMnO_4$ solution needed to be filtered from MnO_2 before use is because, it can distribute the standardization, meaning MnO_2 is insoluble. This process can only be done after the oxidation of the contaminants.

- The reason the titration begins at room temperature is because, the sodium oxalate is very hot and left in the air exposed to acid, which will result in decomposition.

$$C_2O_4{}^{2-}{}_{(aq)} + O_{2(g)} + 2\,H_3O^+{}_{(aq)} \leftrightarrow H_2O_{2(aq)} + 2CO_{2(g)} + 2H_2O_{(l)}$$

$$2H_2O_{2(aq)} \leftrightarrow O_{2(g)} + 2H_2O_{(l)}$$

Procedure:

To begin, a solution was gathered in which 0.02 $KMnO_4$ was prepared a week prior to conduction of this laboratory. The solution was then filtered through a plug of glass wool into a clear 250ml beaker. Next, by adding 55 ml of H_2SO_4 to approximately 900ml of distilled water while stirring a solution of 1M H_2SO_4 is produced. Add a few more milliliters of distilled water till the beaker reaches a desired volume of 1000ml. Next, a dry weighing bottle was gathered and a sample of $Na_2C_2O_4$ was poured into a separate beaker and obtained from the demonstrator. From there, using an analytical balance and the 'weigh by difference technique', three separate 500ml Erlenmeyer flasks of $Na_2C_2O_4$ were accurately weighed to an approximate value between 0.2500 to 0.3000 grams. The three samples recorded were, 0.2614g, 0.2604g, 0.2617g

respectively. Using the 1000ml of the 1.0M $KMnO_4$ produced earlier, add 250ml of the solution to each separate flask and swirl to dissolve. If large chunks are present, use the stirring rod provided to break up contents. Using the burette apparatus available pour the sufficient permanganate to consume about 90-95% of the oxalate, and swirl while doing so until the solution has turned to a colourless substance. While one partner was completing that step, another is to be calculating the accurate permanganate for each flask referring to question 1 of the calculations. After the completion of calculations for all three samples it was determined the volumes of each were 35.11ml, 34.97m, and 35.15ml respectively. Once complete, the hot plate was turned on to a temperature between 55-60°C. The three samples were separately placed on the hot plate for 30 seconds starting with the one with the first pale pink colour. Next the end point of the solution was determined by slowly adding droplets of $KMnO_4$ to the 250ml of 1.0M H_2SO_4 until there is a sign of a pinkish colour. The total volume used was calculated by recording the required volume and subtracting from all of the titrations values and recorded in the data section. The three samples needed 10.4ml, 11.2ml and 9.4ml of $KMnO_4$ respectively. Proceeding to part II, an numbered sample #226 of iron ore was obtained and two separate measurements were distributed using an analytical balance and placed into two seperate 250ml Erlenmeyer flasks. The two weights recorded were, 1.510g and 1.593g respectively. Next, 10ml of 3M of H_2SO_4 and 10ml of H_3PO_4 were added to each separate solution along with a desired amount of distilled water to dissolve the samples. Once completed, using the burette apparatus, each sample was slowly titrated using standardized $KMnO_4$ solution, until the lightest colour of pink appeared after swerling. The two samples used 7.2ml and 6.7ml of $KMnO_4$ to turn the

solution to the slightest bit of pink. Finally, the actual value of iron(II) was obtained from your demonstrator and a substantial amount calculations were formulated using the values recorded.

Observations:

Sample Number: 226- It's formation was in a powdered state with a white colour as its appearance.

The colour of the titration solution was colourless at first but through experimental work and the standardization of $KMnO_4$ it transformed into a light pinkish colour.

The colour of the titration solution at first was also colourless but through the titration of the Fe^{2+} the sample transformed similarly with a pink colour but with a more heavier colour tone

Data:

Unknown Sample: 226
Actual % Value of Fe 2+: 3.53%

Table 1: Sodium Oxalate

	Initial Mass (g)	Final Mass (g)	Mass Sample (g)
Sample 1	13.9337 ± 0.001	13.6724 ± 0.001	0.2614 ± .0002
Sample 2	13.6724 ± 0.001	13.4120 ± 0.001	0.2604 ± .0002
Sample 3	13.4120 ± 0.001	13.1503 ± 0.001	0.2617 ± .0002

Table 2- Part I ($Na_2C_2O_4$ Titration with $KMnO_4$)

	90%			10%			Sample Mass(g)
	Initial Vol	Final Vol	Total Vol	Initial Vol	Final Vol	TotaL Vol	
Sample 1	0.00	37.7	37.7 ± 0.05	37.7 ± 0.05	48.1 ± 0.05	10.4 ± 0.10	0.2614 ± 0.0002
Sample 2	0.00	35.2	35.2 ± 0.05	35.2 ± 0.05	46.4 ± 0.05	11.2 ± 0.10	0.2604 ± 0.0002

Sample 3	0.00	35.5	35.5 ± 0.05	35.5 ± 0.05	44.9 ± 0.05	9.4 ± 0.10	0.2617 ± 0.0002

Table 3- Iron Unknown Sample #226

	Initial Mass (g)	***Final Mass(g)***	***Mass Sample (g)***
Sample 1	17.7450 ± 0.0001	16.2347 ± 0.0001	1.5103 ± 0.0002
Sample 2	16.2347 ± 0.0001	14.6414 ± 0.0001	1.5933 ± 0.0002

Table 4- Part II (Fe^{2+} Titration with $KMnO_4$)

	Sampel Mass	Initial Vol (ml)	Final Vol (ml)	Total Vol (ml)
Sample 1	1.5103 ± 0.0002 g	45 ± 0.05	50 ± 0.05	5.00+2.20=7.20 ± 0.10
		40.2 ± 0.05	42.4 ± 0.05	
Sample 2	1.5933 ± 0.0002 g	42.4 ± 0.05	49.7 ± 0.05	6.7 ± 0.10

Calculations

1. <u>Calculation of 90% of $KMnO_4$ added during Standardization of $KMnO_4$</u>

$$\text{Equation : Vol.Req.90\% Mn}O_4\text{(ml)}=\left(\frac{\frac{Weight\ Na_2C_2O_4(g)}{MW\ Na_2C_2O_4(\frac{g}{mol})}x\frac{2}{5}}{0.02M}\right)\ x\ 0.9x1000ml/L$$

$$Sample\ 1=\frac{\frac{0.2614\ ml}{134\ g/mol}\times\frac{2}{5}\times 0.9\times 1000ml/l}{0.02}$$

=35.08 ml

$$Sample\ 2=\frac{\frac{0.2604\ ml}{134.1\ g/mol}\times\frac{2}{5}\times 0.9\times 1000ml/l}{0.02}$$

=34.95 ml

$$Sample\ 3=\frac{\frac{0.2617\ ml}{134.1\ g/mol}\times\frac{2}{5}\times 0.9\times 1000ml/l}{}$$

$$\overline{0.02}$$

=35.13 ml

2. Calculation of [KMnO4]

Sample 1= $\frac{(0.2614g / 134.1g/mol \; x \; \frac{2}{5}}{(48.1 \; ml \; /1000 \; ml/l)} = 0.0162$ mol/l

Sample 2= $\frac{(0.2604g / 134.1g/mol \; x \; \frac{2}{5}}{(46.4 \; ml \; /1000 \; ml/l)} = 0.0167$ mol/l

Sample 3= $\frac{(0.2617g / 134.1g/mol \; x \; \frac{2}{5}}{(44.9 \; ml \; /1000 \; ml/l)} = 0.0174$ mol/l

Percent Uncertainty

$$= \sqrt{\frac{sum \; of (M_{avg} - M_i)^2}{N-1}}$$

$$= \sqrt{\frac{(0.0168-0.0162)^2}{3-1} + \frac{(0.0168-0.0167)^2}{3-1} + \frac{(0.0168-0.0174)^2}{3-1}}$$

= 6.04E-4

Uncertainty

$$= \frac{6.04E-4}{0.0168} \text{ x } (100) = 3.6\%$$

Relative Spread

$$= \frac{highest \; unknown - lowest \; unknown}{average \; unknown} x(1000)$$

$$= \frac{0.0174-0.0162}{0.0168} \; x \; 1000 \; = 71 \; ppt$$

3. Calculation of Fe2+

Equation $= \frac{(Vol.KMnO_4/1000mL/L)(Av.[KMnO_4])(MW\ Fe)(5)}{mass\ Fe}(100)$

Sample 1 $= \frac{(7.2mL/1000mL/L)(0.0168)(55.845g/mol)(5)}{1.5103}(100) = 2.24\%$

Sample 2$= \frac{(6.7mL/1000mL/L)(0.0168)(55.845g/mol)(5)}{1.5933}(100) = 1.97\%$

Average Fe^{2+}

$= \frac{(2.24)+(1.97)}{2} = 2.11\%$

Percent Uncertainty:

$= \sqrt{\frac{sum\ of(M_{avg}-M_i)^2}{N-1}} = \sqrt{\frac{(2.12-2.24)^2}{2-1} + \frac{(2.12-1.97)^2}{2-1}} = 0.19$

$= \frac{0.19}{2.11}x(100) = 9\%$

Relative Spread

$= \frac{highest\ unknown - lowest\ unknown}{average\ unknown}(1000)$

$= \frac{2.24-1.97}{2.11}(1000) = 128$ ppt

Percent Error

$\frac{|experimental - theoretical|}{theoretical}(100)$

$= \frac{|2.11-3.53|}{3.53}(100) = 40.22\%$

Discussion

After the completion of this lab, the experimental value of Iron content was lower than the theoretical percentage of Iron content. There are a variety of reasons for the discrepancies between the two values mainly due to experimental and chemical error. Firstly, during the duration of this lab, some of the Iron could have possibly reacted with contaminants instead of permanganate which would result in a smaller amount of permanganate needed to titrate the rest of the iron. Secondly, during titration, some permanganate content was stuck to the sides of the flask. This will result in a value higher then it should be, therefore lowering the % of Iron. Lastly, it is possible that the mixture was not homogeneous and did not fully mix with the iron contents resulting in a lower volume. Subsequently all of these reasons could have potential altered results and therefore cause the experimental percent value of Iron to be lower than its real value.

Conclusion

In conclusion, using redox-oxidation titration, the Iron % content of Fe^{2+} was determined in an unknown sample #226, in which was concluded to have an average percent mass value of 2.11%. This value for our experiment was calculated to be lower than its actual value of 3.53%. It was also determined that it's percent uncertainty for $KMnO_4$ and Fe^{2+} were 3.6% and 9.0% respectively. Lastly, its percent error was calculated to be 40.2% along with a relative spread of 128ppt.

Bibliography

CHEM 1001/1005 CHEM 1002/1005 “Introductory Chemistry Laboratory Manual”. ‘Robert . Burk’, M. Azad, X.Sun, P.A Wolf. Carleton University,2018-2019.