EXAMPLES

TION OF POTASSIUM HYDROGEN PHTHALATE
ID UNKNOWN SODIUM CARBONATE

Quantitative Analysis

Section: 041

October 10, 2002

Date lab completed : 10/1/02

Unknown # 13

Introduction

The titration for this lab was a strong acid to strong base type titration also called a neutralization type titration. The strong base, NaOH was the titrant and the strong acid, HCl was the analyte for determining the base/acid ratio. The chemical equation for the ions in the titration is seen below in reaction 1. For this type of titration a acid base type indicator is used as opposed to a base acid type indicator. An acid base indicator is a weak organic acid whose undissociated form differs in color from its conjugate base form as seen in the chemical equilibrium equation below in reaction 2.

$$H_3O^+ + OH^- \rightarrow 2H_2O \quad (1)$$

$$HIn + H_2O \rightarrow H_3O^+ + In^- \quad (2)$$

The indicator will have a complete color change in the transition range ph for that particular Indicator. The indicator used for this lab was phenolphthalein which as a transition range pH of 8.3 to 10.0 and its color change is from colorless to pink.

At the start of the titration, the analyte solution pH is that of the strong acid.

$$pH = -\log [H_3O^+] = -\log [.1M] = 1$$

The equivalence point of the titration occurs when there are equal amounts of strong acid and strong base, therefore the pH at that point will be that of water, pH = 7.00. Since the transition range of the indicator is above a pH of 7.00 we can say for certain that the end point occurs soon after the equivalence point.

After the base acid ratio is found, the NaOH is standardized with KHP in the reaction 3 and the unknown sodium carbonate is determined from HCl as the titrant in reaction 4.

$$KHC_8H_4O_4 + NaOH \rightarrow KNaC_8H_4O_4 + H_20 \quad (3)$$

$$Na_2CO_3 + 2HCl \rightarrow H_2CO_3 + 2NaCl \quad (4)$$

Table 2: Determination of unknown #13 weight %

run	mass unknown (g)	volume HCl titrated (L)	mass Na_2CO_3 (g)	wt % Na_2CO_3
1	0.834	0.0425	0.567	67.99
2	0.827	0.0450	0.598	72.31
3	0.856	0.0410	0.543	63.43
				wt % avg = 67.91

Table 3: Group #1 Determination of unknown # 13

Chemist	Concentration of titrant (M HCl)	average wt % of Na_2CO_3 in unknown sample
[redacted]	0.0999	67.43
[redacted]	0.0987	73.05
[redacted]	0.1026	65.43
[redacted]	0.0975	70.98
		wt % avg = 69.22

Statistics

Standard Deviation:

[redacted]: SD = .234 RSD = 2%

Group: SD = .876 RSD = 7%

Confidence limits: $CL = x \pm (ts) / \sqrt{N}$

[redacted]: $67.91 \pm (3.18 * .234) / \sqrt{3} = 67.91 \pm .43$

Group: $69.22 \pm (3.18 * .234) / \sqrt{4} = 69.22 \pm 1.39$

Conclusion

The trials for my unknown determination and for the group as a whole were very precise with RSDs around 5% meaning that all the data for the trials were within 5% of each other. Furthermore, I am 95% confident that the percent of Na_2CO_3 in unknown #13 was between 68.34% and 67.48% while the group has a 95% confidence between 70.61% and 67.83%.

Reagents

DI water	.1M HCl	Phenolphthalein
12M HCl	.1M NaOH	Na_2CO_3 Unknown #13
NaOH pellets	KHP	

Solution Preparations

1L of .1M HCl: $M_1V_1 = M_2V_2$; (.1M)(1000ml) = (12M)V2 ; V2 = 8.33ml HCl in 1L DI water

1L of .1M NaOH : $\frac{.1 \text{ mol NaOH}}{\text{L}} * \frac{40 \text{ g NaOH}}{\text{mol}} * 1 \text{ L} = 4\text{g NaOH}$ in 1L DI water

Results

Standardization of Titrant:

$$\text{M NaOH} = \text{x mass KHP} * \frac{\text{mol KHP}}{204\text{g KHP}} * \frac{1 \text{ mol NaOH}}{1 \text{ mol KHP}} * \frac{1}{\text{L NaOH}}$$

Table 1: NaOH standardization

run	mass KHP(g)	volume NaOH titrated (L)	NaOH concentration (M)
1	0.726	0.0351	0.1014
2	0.743	0.0370	0.0984
3	0.735	0.0360	0.1001
			M avg = .0999

Determination of unknown:

M HCl = M NaOH * (base-acid ratio) ; base-acid ratio = 1 ; M HCl = .0999mol/L

$$\text{X mass of } Na_2CO_3 = \text{L HCl} * \text{M HCl} * \frac{1 \text{mol } Na_2CO_3}{2 \text{ mol HCl}} * \frac{\text{g } Na_2CO_3 \text{ (unknown)}}{1 \text{ mol } Na_2CO_3}$$

$$\text{wt \% } Na_2CO_3 = \frac{\text{X mass } Na_2CO_3 \text{ (100)}}{\text{mass unknown sample}}$$

SPECTROMETIC DETERMINATION OF IRON

M

Quantitative Analysis Lab – Spring 2009

Section: 002

3.18.09

Introduction

The purpose of this lab is to determine what the concentration of iron is in an unknown solution. This is done by first preparing several solutions with different concentrations of iron and determining the absorbance of each solution. These values can then be plotted because absorbance increases linearly as concentration increases. The absorbance of the unknown solution can then be measured, and based on the equation of the linear regression fit, the graph can then be extrapolated and solved for the concentration of the unknown sample.

When preparing the standard solutions, the exact concentration of all of the components must be known so that the correct calibration curve is created. Ideally, exact concentrations will result in a perfectly linear line because according to the Beer-Lambert law, the fraction of incident light absorbed is directly proportional to the concentration of the sample. Specifically, Beer-Lambert law states $A = \varepsilon b C$, with A being the absorbance, ε being the molar absorptivity constant, b being the length of the cuvette used, and C is the concentration of the solution.

Nonetheless, the law has several limitations and the true value of concentration can deviate from the actual concentration in several instances. When the concentration of the solution is too high, it should result in a high absorbance value. However, the spectrometer can't reliably read high absorbance values, so high concentrations should be avoided in this experiment. In addition to yielding an unreliable absorbance reading, the reading can be affected due to intermolecular interactions when the concentration of the iron is high. The results will also be unreliable if the solution is not homogeneous throughout and there are suspended particles that might cause light scattering. As always, inaccurately calibrated equipment may also result in inaccurate results.

The addition of hydroxylamine reduces the iron solution from Fe^{3+} to Fe^{2+}, and the orthophenanthroline (phen) binds to the iron to form an orange-colored complex.

$$2\ Fe^{3+} + 2\ NH_2OH + 2OH^- \rightarrow 2\ Fe^{3+} + N_2 + 4H_2O$$

$$Fe^{2+} + 3\ phen \rightarrow Fe(phen)_3^{2+}$$

The more iron there is in the solution, the darker the solution is and will absorb more light. The spectrophotometer is used to determine how much light is absorbed. The spectrophotometer has two components to it, one that produces light of selected wavelengths, and one that measures the intensity of light absorbed. The solution sample is placed into a cuvette in between the two components. The same cuvette and same machine must be used to reduce instrumental error. Every cuvette will be slightly different, and one must use a blank sample (with no iron) to determine the zero value of absorbance and calibrate the machine to that value. The spectrophotometer can be set to a specific wavelength of light it releases. We are using 510 nm because that is the wavelength at which the most absorbance of the iron complex occurs. A single beam of light is released and passes through the cuvette and the solution sample. The intensity of light not absorbed by the solution is measured by the machine. The more concentrated the solution is the darker the solution is, and the higher the absorbance is.

When the absorbances of several different concentrations are measured, they should be plotted on a graph to produce a calibration curve. This should be a linear line, but at high concentrations, the absorbance will eventually turn into an exponential equation and even out. The point of maximum concentration at which the absorbance no longer fits a linear line is known as the linear dynamic range, and concentrations that do not produce a linear line should

not be used. Ideally, the points should form a perfectly linear line. However, this does not always happen. As a result of this, the best fit line should be used, which is known as the linear regression fit. The best fit line is defined as one that minimizes the sum of deviations from all the data points to the line. The measure of goodness of fit is displayed by the r^2 value, with $r^2 = 1.0$ displaying perfect fit and a value of $r^2 = 0.0$ being a line of no fit. This line can then be used to predict values of either value when one knows the other through the equation of the line. The closer r^2 is to 1.0, the more confident one can be of their predicted value.

If one does not have a linear line for the calibration curve, or the r^2 value is extremely low, the experiment should be done again with higher precision in order to reduce experimental error. If repeating the experiment still does not yield a linear line, lower concentrations of iron should be used so as to increase the accuracy of the spectrophotometer.

References:

- Spring 2009 Lab manual
- "Spectrophotometric Determination of Iron" additional reading
- http://sbio.uct.ac.za/Sbio/postgrad/modules/GRD/spectrophotometry/beer1.php
- http://hplc.chem.shu.edu/NEW/HPLC_Book/Detectors/det_dynr.html
- http://www.curvefit.com/linear_regression.htm
- http://www.ruf.rice.edu/~bioslabs/methods/protein/spectrophotometer.html

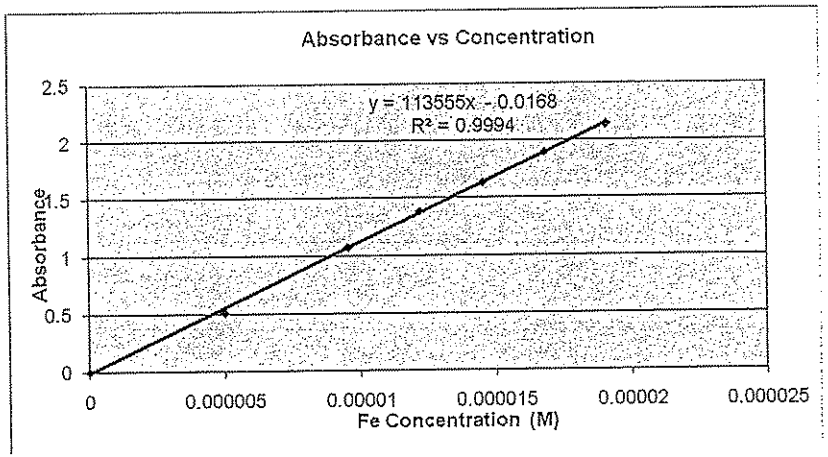
Reagents

- Hydroxylamine hycrochloride reductant
- Sodium acetate buffer (pH = 8)

Results

Table 2: Absorbance Readings for Standards

Standard	Concentration	Absorbance
1 (blank)	0 M	0
2	4.97×10^{-6} M	0.511
3	9.55×10^{-6} M	1.073
4	1.22×10^{-5} M	1.387
5	1.45×10^{-5} M	1.632
6	1.68×10^{-5} M	1.898
7	1.91×10^{-5} M	2.142



According to the graph, the best fit line has the following equation:

$$(absorbance) = 113555(Fe\ Concentration) - 0.0168$$

Absorbance of unknown solution C: 1.249

To calculate the concentration of our unknown, the equation of the best fit line can be rearranged:

- Orthophenanthroline
- 1.91×10^{-4} M standard iron solution
- Unknown # C

Solution Preparation

First, standards have to be made in order to create a linear dynamic range. 7 of these standards were made, which include a blank standard. Into each flask, 1 mL of hydroxylamine hycrochloride reductant, 5 mL of sodium acetate buffer and 5 mL of orthophenanthroline were placed into each flask, along with different amounts of standard iron solution, ranging from none (blank) to 5 mL. The solution was then diluted to 50.0 mL, and the molarities of each solution were then calculated.

$$M_1V_1 = M_2V_2$$

$$Molarity\ of\ standard = \frac{(Molarity\ of\ Fe)(volume\ of\ Fe\ added)}{(total\ volume\ of\ standard\ solution)}$$

For standard 3:

$$\frac{(1.91 \times 10^{-4}\ M)(2.50\ mL)}{(50.0\ mL)} = 9.55 \times 10^{-5} M$$

Table 1: Preparation of Standard Solutions

Standards	Amount of hydroxylamine hycrochloride reductant used	Amount of sodium acetate buffer used	Amount of orthophen-anthroline used	Amount of stock Fe solution used	Calculated Molarity
1 (blank)	1.00 mL	5.00 mL	5.00 mL	0.00 mL	0 M
2	1.00 mL	5.00 mL	5.00 mL	1.30 mL	4.97×10^{-6} M
3	1.00 mL	5.00 mL	5.00 mL	2.50 mL	9.55×10^{-6} M
4	1.00 mL	5.00 mL	5.00 mL	3.20 mL	1.22×10^{-5} M
5	1.00 mL	5.00 mL	5.00 mL	3.80 mL	1.45×10^{-5} M
6	1.00 mL	5.00 mL	5.00 mL	4.40 mL	1.68×10^{-5} M
7	1.00 mL	5.00 mL	5.00 mL	5.00 mL	1.91×10^{-5} M

$$(Fe\ concentration) = \frac{(absorbance) - 0.0168}{113555}$$

For unknown C, the iron concentration can be determined to be:

$$\frac{1.249 - 0.0168}{113555} = 1.115 \times 10^{-5} M$$

Conclusion

The purpose of this experiment was to determine what the concentration of iron is in unknown sample C. This took a while to do, because according to the original lab manual, we were supposed to use four times the amount that we ended up using in the end. The original concentrations that were to be used did not yield in accurate absorbances, because the line was not linear. However, with the concentrations that we did use resulted in a linear regression line with a goodness of fit calculation of 0.9994. According to this data, the determined concentration of iron in the sample can be calculated to be 1.115×10^{-5} molar with great certainty. There are several reasons that could result in the measured concentration being inaccurate. The greatest degree of error comes from different individuals performing the experiment. This increases human error because not everyone measures the solutions the same way, and we cannot be sure that the other people are being as accurate as they should be. Another error source could be the machine not working correctly and thus not recording the right absorbance. An additional reason why our calculated concentration could be mistaken would be if the stock solution of iron was not correctly calculated or if the stock solutions of any of the other reagents used in this experiment were not the concentration that we thought they were. While we would still get a linear regression line, the calculated concentration would not be what it really is, though the only way to test this would be to perform the experiment again, assuming that the stock solutions used in this trial were made correctly.