Titrimetric Determination Abdul Fayeed Abdul Kadir, Partner: Linh Nguyen October 1st, 2019

Summary Report

The objective of this experiment was to determine the concentration of the two unknown acetic acid samples. This was done by reacting sodium hydroxide with the acetic acid samples. The sodium hydroxide was prepared by adding approximately 6 mL of its stock solution into a 1-liter bottle and was mixed with distilled water up to the shoulder of the bottle. The sodium hydroxide solution was then titrimetrically transferred into a known mass of KHP (potassium hydrogen phthalate) in the Erlenmeyer flask. The mass of each dried KHP sample was ensured to be within 0.4-0.7 g before it was dissolved with approximately 30 mL of distilled water in the flask. About 2-3 drops of phenolphthalein was added into the flask before the titration began and the sodium hydroxide solution was titrated to the end point, where the solution turned to faint pink from colorless solution. The titrated volume was calculated, and 3 replicate measurements were obtained to calculate the average concentration of sodium hydroxide solution.

The reaction of sodium hydroxide solution with the KHP solution was stoichiometrically 1:1 mole ratio. To simplify, the H⁺ ions from KHP reacted with the OH⁻ ions from sodium hydroxide solution by 1:1 mole ratio. From that relationship, the mole of NaOH reacted can be determined by calculating the mole of KHP solution first. The calculation was done in excel, by referring to **Table 1** below. The formula used to calculate the values in Table 1 was as shown in **Table 2** below. The **average molarity of sodium hydroxide solution** was found to be **0.1077 M.**

Table 1: Molarity of NaOH for each sample			
Sample #	1	2	3
Mass (g)	0.6590	0.5274	0.6664
Initial buret reading (mL)	0.60	0.03	0.70
Final buret reading (mL)	30.62	24.05	30.91
Titrated volume (mL)	30.02	24.02	30.21
Molar mass KHP (g/mol)	204.22		
Mole of KHP (mol)	0.003227	0.002583	0.003263
Mole of NaOH (mol)	0.003227	0.002583	0.003263
Molarity NaOH (M)	0.1075	0.1075	0.1080
Average molarity NaOH (M)	0.1077		
Standard deviation NaOH (M)	0.0002958		
Relative standard deviation NaOH (%)	0.2747		

Table 2: Formula used to calculate values in Table 1		
Formula		
Titrated volume (mL)	Final buret reading (mL) - Initial buret reading (mL)	
Mole of KHP (mol)	Mass (g) / Molar mass (g/mol)	
Mole of NaOH (mol)	Same as above since 1:1 ratio	
Molarity of NaOH (M)	Mole of NaOH (mol) / Titrated volume (L)	
Average molarity NaOH (M)	AVERAGE(values) in excel	
Standard deviation NaOH (M)	STDEV(values) in excel	
Relative standard deviation NaOH (%)	[Standard deviation NaOH (M) / Average molarity NaOH (M)] x 100 %	

Next, the average concentration of sodium hydroxide solution was used to determine the concentration of each acetic acid sample. 25 mL of each sample were transferred by pipette into a 250 mL volumetric flask and diluted, and 25 mL of the diluted sample was transferred by pipette into the Erlenmeyer flask. The sodium hydroxide solution in a burette was titrimetrically transferred into each acid sample in the flask, that has been mixed with 2-3 drops of phenolphthalein, to the end point, where the solution turned faint pink from colorless solution. Three replicate measurements of titrated volume were obtained for each sample and the concentration of each sample was calculated and averaged. The first three flasks were for sample 1 and the next three flasks were for sample 2 of acetic acid. The concentration of the stock solution was determined by taking into account the dilution factor to be pooled with the entire class data and analyzed.

Same as before, the reaction of sodium hydroxide solution with the acetic acid stoichiometrically was 1:1 mole ratio. In other words, the H⁺ ions from acetic acid reacted with the OH⁻ ions from sodium hydroxide solution by 1:1 mole ratio. The mole of sodium hydroxide titrated into each flask was determined and the molarity of the acetic acid of each sample was calculated. The concentration of stock acetic acid was determined by taking into account the dilution factor of 10, as it was diluted previously before it was titrated with the sodium hydroxide solution. These calculations were made in excel as shown in **Table 3** and the formula used were as listed in **Table 4** below. The **average molarity of sample 1 acetic acid** was **0.8289 M** while the **average molarity of sample 2 acetic acid** was **1.860 M**.

Table 3: Molarity of sample 1 and 2 of acetic acids in flasks 1-3 and 4-6 respectively						
Flask #	1	2	3	4	5	6
Initial volume in flask (mL)	25.00	25.00	25.00	25.00	25.00	25.00
Initial buret reading (mL)	0.51	19.78	0.52	0.30	0.49	0.18
Final buret reading (mL)	19.78	39.02	19.75	43.49	43.65	43.40
Titrated volume (mL)	19.27	19.24	19.23	43.19	43.16	43.22
Average molarity NaOH (M)	0.1077					
Mole of NaOH (mol)	0.002075	0.002072	0.002071	0.004650	0.004647	0.004654
Mole of CH3COOH (mol)	0.002075	0.002072	0.002071	0.004650	0.004647	0.004654
Diluted molarity CH3COOH (M)	0.08300	0.08287	0.08282	0.1860	0.1859	0.1861
Dilution factor	10	10	10	10	10	10
Original molarity CH3COOH (M)	0.8300	0.8287	0.8282	1.860	1.859	1.861
Mean molarity CH3COOH (M)	0.8289			1.860		
Standard deviation CH3COOH (M)	0.000897		0.001292			
Relative standard deviation						
снзсоон (%)	0.1082		0.06946			

Table 4: Formula used to calculate the values in Table 2		
Formula		
Titrated volume (mL)	Final buret reading (mL) - Initial buret reading (mL)	
Mole of NaOH (mol)	Volume (L) x Average Molarity NaOH (M)	
Mole of CH3COOH (mol)	Same as above since 1:1 ratio	
Diluted molarity CH3COOH (M)	Mole of CH3COOH (mol) / Initial flask volume (L)	
Dilution factor	250 mL / 25 mL	
Original molarity CH3COOH (M)	Diluted molarity x Dilution factor	
Mean molarity CH3COOH (M)	AVERAGE(values) in excel	
Standard deviation CH3COOH (M)	STDEV(values) in excel	
Relative standard deviation		
СН3СООН (%)	[Standard deviation CH3COOH (M) / Mean molarity CH3COOH (M)] x 100 %	

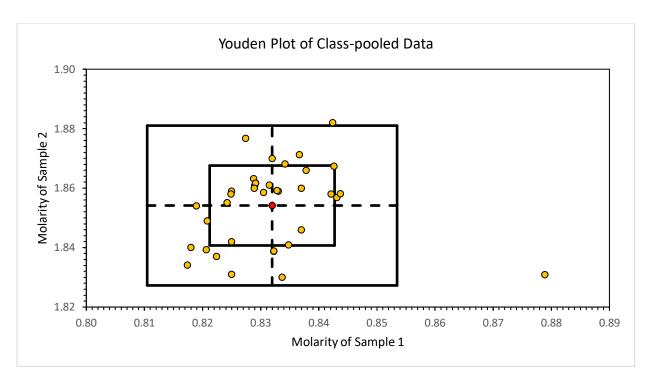


Figure 1: Youden Plot of Class-pooled Data

Construction of Youden Plot

A Youden Plot of class-pooled data was plotted as shown in Figure 1 above. Before plotting the Youden Plot with the class-pooled data, the outliers of each sample were removed. The first method was to remove the outliers by visual inspection. Some of the values that looked obviously out of scale from the other values (example: ~0.008 from a set of values of ~0.08) were removed immediately without conducting any test to prove their outliers' properties. This method was shown in **Table 5** and **10** for sample 1 and 2 respectively. The data removed were highlighted in yellow. The second step was to calculate the Student's t-value of the mean of each sample. The Confidence Interval (CI) used for both samples were t-value at CI of 99 %. Any values that fall outside the lower and upper limits of the mean were removed. This step was shown in **Table 6** and 11 for sample 1 and 2 respectively (data removed that were highlighted yellow). The values of upper and lower limit of the mean with the Student's t-test value were calculated in excel, as tabulated in Table 7 and 12 for sample 1 and 2 respectively by using the formula listed in Table 9 and 14 respectively. The removal of outliers by Student's t-value was only done once, although, theoretically more iterations needed until there were no more outliers left. The main reason why only one iteration was needed because only less than 10 data points left if further iterations were done, which were not enough for the analysis of the plot. The remaining data that lie within the mean for each sample were as tabulated in **Table 8** and **13** for sample 1 and 2 respectively. **Table** 5 - 14 can be found in the Excel file attached together with this report.

By cross-examining the data in **Table 8** and **13**, the values that do not have their pairs from the other sample were removed, as highlighted in yellow in both tables. The final data for the Youden Plot were compiled, as shown in Table **15**. The removed outliers were grouped and tabulated as shown in **Table 19**, **20** and **21** (written description in the table for which data removal).

The mean and standard deviation of each sample were calculated as tabulated in **Table 16**. The formula used to find the mean and standard deviation were the same as listed in **Table 9/14**. To plot the boxes of standard deviation in the plot, the data were calculated as shown in **Table 17a** and **b**, for 1 and 2 standard deviation respectively. Meanwhile, the vertical and horizontal dashed line in the Youden plot, passing through the mean point, were calculated and drawn by using the points in **Table 18a** and **b** respectively. **Table 5-21** can be referred to from the Excel file attached together with this report.

Analysis of Youden Plot

Based on the Youden plot, most of the data points lay within the boxes of 2 standard deviations. Only two points lay outside of the boxes. This indicated that the two values deviated by a significant amount from the mean of the molarity of acetic acid of both samples. The rightmost point had an approximate molarity of 1.83 M of sample 2, which did not deviate too far from the mean of sample 2. However, its molarity of sample 1 deviated by a significant amount from the sample 1's mean, with a molarity of approximately 0.879 M. The other way around went for the topmost point, where it deviated much from the mean for the sample 2's molarity, but not for the sample 1's molarity. This might happen due to rounding errors made by the students when calculating the molarity of each sample. Other than that, the values were too small/large due to the fact that the molarity of acetic acid was determined by using the molarity of sodium hydroxide solution calculated in the first half of the experiment, where it would affect the entire calculation if errors had been made during the first half.

Within the boxes of standard deviation, most of the data points lay within the +x, +y and +x, -y quadrants. Each quadrant of the boxes represented different things. Based on the plotted data points, the points that fell within the +x, +y quadrant represented positive systematic errors, where students tended to report results higher than what they supposed to be for both samples. The total opposite explanation for data points that fell within -x, -y quadrant, which was called as negative systematic errors. Meanwhile, the data points within the +x, -y quadrant represented the values where the students reported higher values for sample 2 but lower values for sample 1, than what they supposed to have in nature. Consequently, the data points that fell within the -x, +y quadrant were the values students reported which were higher for sample 1 but lower for sample 2, than what it actually measured.

Generally speaking, the data points were evenly split to deviate from the mean by 1 and 2 standard deviation (half deviates from the mean by 1s, the other half deviates from the mean by 2s). Only a few points that were actually really closed to the mean of both samples, which indicated less significant size of errors made during the experiment by the students. However, most of the students did make significant amount of errors that deviated their calculated values from the mean. By using this Youden plot, what I can learn from it is that I am able to predict how the data deviates from the mean of each axis value easily (in this case, the molarity of the two samples acetic acid). Also, each quadrant in the plot shows different type of systematic errors, that can be analyzed and inferred.