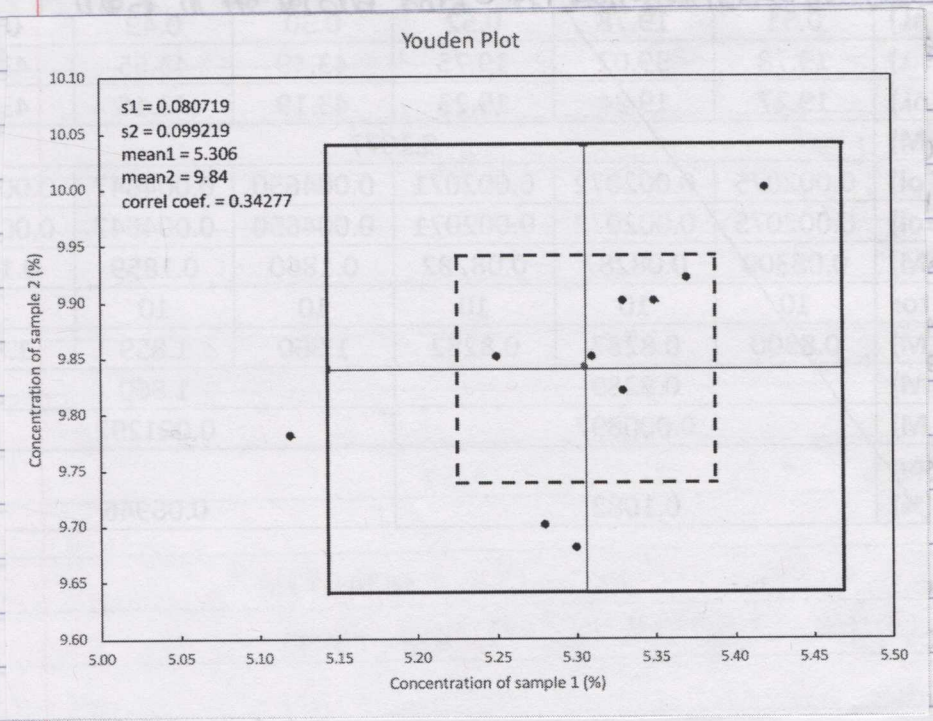
Lab 3
Titrimetric Determination
10/22/2019

Partner: Linh Nguyen

Sample number	1	2	3	4	5	6	7	8	9	10
Control sample 1	5.33	5.42	5.12	5.25	5.35	5.37	5.33	5.31	5.28	5.30
Control sample 2	9.90	10.00	9.78	9.85	9.90	9.92	9.82	9.85	9.70	9.68

Mean 1	5.31
Mean 2	9.84
1 Standard deviation (sample 1)	0.0807
2 standard deviation (sample 1)	0.1614
1 standard deviation (sample 2)	0.0992
2 standard deviation (sample 2)	0.1984



→this lab's prelab is at page 53, which is further away from its lab description, due to the fact that this prelab is done and not printed & pasted before coming to the lab, since at the moment, we can just show the graph from our computer for checking purposes by the TA.

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10/1/2019

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Objective

The purpose of this experiment is to titrimetrically determine the concentration of acetic acid in 2 vinegar samples.

Introduction

A Youden plot is introduced, where it ~~is~~ shows a plot of two control samples on both axis, of a given set of measurement in pair. Two control samples ~~is~~ are to be analyzed and the class data are pooled to plot the Youden Plot. By referring to the plot, two boxes are drawn around the mean of both samples; smaller box encompasses the single standard deviation of each axis and the bigger box encompasses the double standard deviation of each axis. A set of data that falls in the +x & +y quadrant of the box is said to have a positive systematic error in the analysis. Data that are in the -x & -y quadrant is said to have a negative systematic error.

Apparatus

- 6 – 250mL Erlenmeyer flasks
- 1 – 50mL buret
- 1 – 10mL graduated cylinder
- 1 – 100mL graduated cylinder
- 1 – 25mL pipet.
- 1 – 1-L polyethylene bottles/glass bottles with screw caps.

Chemicals

- phenolphthalein solution (0.1% in ethanol)
- KHP solid, previously dried
- 50% NaOH solution (50g NaOH in 50mL H_2O)
- vinegar control sample (~5% (w/w) acetic acid)
- vinegar ~~sa~~ control sample 2 (~10% (w/w) acetic acid).

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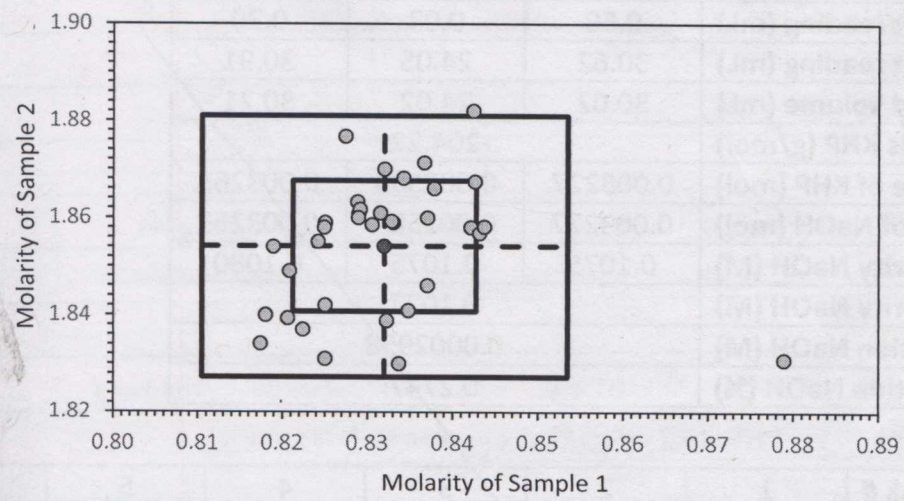
KHP Samples

Sample #	1	2	3
Mass (mg)	~~0.6590~~ ~~0.4637~~	0.5274	0.6664
Initial reading of buret (mL)	0.60 ~~0.28~~ ~~0.60~~ 30.62	0.03	0.70
Final reading of buret (mL)	30.62	24.05	30.91
Titrated volume (mL)	30.02	24.02	30.21

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		



Youden Plot of pooled data for acetic sample 1 & 2's concentrations.

→ mol of KHP to mol of NaOH is 1:1 ratio

→ mol of KHP = $\frac{\text{mass}}{\text{molar mass}}$

→ molarity NaOH = $\frac{\text{mol NaOH}}{\text{titrated vol (L)}}$

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Procedure

1. Add ~ 6 mL of 50% NaOH stock solution to a clean 1-L bottle (use 10-mL graduated cylinder).

2. Weigh 3 0.4–0.7g samples of dried KHP to the nearest 0.1 mg.

3. Place each sample in a labeled 250-mL Erlenmeyer flask.

4. Add about 30 mL DI water, dissolve the KHP.

5. Add 2/3 ~~drops~~ drops of phenolphthalein solution to each flask.

6. Fill the 50-mL buret with NaOH solution & make initial buret reading.

7. Titrate KHP solution in the first flask to the end point. (at the endpoint, the color changes from colorless to faint pink that persists ~ 1 minute, then fades)

8. Record the final volume.

9. Repeat step 5 to 7 for each of the other KHP samples.

10. Place a rubber stopper / lid on the bottle of NaOH solution ~~of~~ label with the contents, date & names of the group and lab section.

11. Use a pipet to deliver 25.00mL of acetic acid control sample 1 into a labeled / 250 volumetric flask.

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Acetic Acid Control Samples

Flask #	1	2	3	4	5	6
Initial volume in the Erlenmeyer flask (mL)	25.00	25.00	25.00	25.00	25.00	25.00
Initial reading of buret (mL)	0.51	19.78	0.52	0.30	0.49	0.18
Final reading of buret (mL)	19.78	39.02 ~~26.42~~	19.75	43.49	43.65	43.40
Titrated volume (mL)	19.27	19.24	19.23	43.19	43.16	43.22

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 CH3COOH (%)	0.1082			0.06946		

→ mol NaOH : mol CH_3COOH 1 : 1 ratio

→ diluted molarity CH_3COOH = $\frac{\text{mol } CH_3COOH}{\text{mL in flask initially (L)}}$

→ original molarity CH_3COOH = diluted molarity · $\left(\frac{250\text{mL}}{25\text{mL}}\right)$

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12. Dilute the solution to the mark & mix very well (stopper and invert the solution in the flask at least 15x)

13. Use a pipet to deliver 25.00 mL of solution into each of 3 clean 250-mL Erlenmeyer flasks, and labeled 1, 2 and 3.

14. Repeat step 11-13 for control sample 2, labeling the flasks 4, 5 & 6.

15. Add 2 drops of phenolphthalein solution to each of the 6 Erlenmeyer flasks.

16. Fill a 50-mL analytical burette with a standard NaOH solution.

17. Read the initial volume and titrate the solution in each of the flasks to the end point (colorless to pink color).

18. Record the end-point volumes to the nearest 0.01mL.

19. Store NaOH standard as advised, then dispose the titration solution & other solutions as indicated

20. Clean & dry the lab work area and upload the data before leaving the lab.

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Analysis & Results

All of the analysis and related table and graphs are included together with the summary report of this lab. Some important table and graphs are posted in the previous pages, illustrating the calculated value of the concentration of NaOH and both sample of acetic acid. The explanation on calculation of each reaction is provided under each table.

A Youden Plot is graphed by using the class-posted data. After the removal of outliers (which will be provided in the PDF), the graph pasted in page 21 is obtained. Most of the data lie in between the ±2s of the mean of data. Only 2 of them fall out of the ± 2 standard deviation. By looking at the +x,+y quadrant of both ± 1s & ±2 s, the data obtained from a ~~sys~~ positive systematic error in the analysis, where the results reported are too high than what it is supposed to be. It is the opposite for data at the -x,-y quadrant. At either quadrant +x,-y or -x,y, the data indicate that either the concentration of sample 1 or 2 is reported to be lower ~~at~~ or higher than what it supposed to be, but not both (1 higher, 2 lower, vice versa).

Conclusions

The molarity of NaOH obtained from the three KHP samples are fairly around the same value. This is also the same for the acetic acid sample. Most of the class-posted data fall within the mean of the data, same goes to my obtained concentrations.