Experiment 4: Titration

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Introduction:

In this experiment, the titration of a known base is used to identify an unknown acid

using acid-base titration methods. The acid $NaOH$ is diluted down, standardized with a known monoprotic acid, and then used to find the concentration of an unknown diprotic acid. The standardization of the base ensures the concentration of base is accurate to 3 significant digits to allow for maximum precision in the standardization of the unknown diprotic acid. The acid-base titration with the unknown diprotic acid is done in three trials, after which the equation $c❑_{base} gV❑_{base} = \frac{b}{a} c❑_{acid} gV❑_{acid}$ is used to find the concentration of the unknown acid. The equation $C❑_1 V❑_1 = C❑_2 V❑_2$ is also used to convert between the concentrations of the acids and the base, and the concentrations of their respective solutions.

Materials:

1. 4.00 mL 6 mol/L NaOH
2. 150 mL beaker
3. 30 mL Standard Acid solution
4. 30 mL Unknown Acid
5. pH probe
6. Drop counter
7. Stirrer
8. Phenolphthalein
9. 25 mL buret
10. Plastic funnel
11. 50 mL beaker
12. 400 mL beaker
13. 10 mL graduated cylinder (+/-0.001 mL)
14. Stirrer plate

Procedure:

1. Rinse a 400 mL beaker with distilled water and dry it
2. Use a 10 mL graduated cylinder to measure approximately 4-5 mL of concentrated NaOH. Record this approximate value and the concentration of the NaOH as written. NOTE: DO NOT obtain the NaOH in a graduated cylinder! Use a small beaker and share it with the people in your work area!!!
3. Add approximately 245 - 250 mL of distilled water to the 400 mL beaker. Record this approximate value
4. Add the concentrated NaOH to the water while stirring. Continue to stir for approximately 2 min
5. Pour the excess concentrated NaOH solution in the waste container (if nobody else in your area can use it!), then rinse the container well.
6. Turn on the LabQuest 2
7. Attach the Drop Counter to an L bar
8. Connect the Drop Counter to the DG1 port of the LabQuest 2
9. Use a universal clamp to secure the plastic buret above the detector of the Drop Counter. The buret has 2 taps. Close both taps fully (they should be horizontal)
10. Fill the plastic buret to the 40 mL mark with your dilute NaOH solution (from step 4), then

place a 10 mL graduated cylinder under the tip (and also below the detection region of the Drop Counter)

11. Open the bottom tap on the plastic buret completely and then slowly open the top tap until the dilute NaOH is dropping at a rate of 1-2 drops/second. NOTE: Be careful to align the tip of the buret above the CENTER of the detector on the Drop Counter. The drops should not be dropping onto the Drop Counter, but through the center of the C. Also ensure that the red light is FLASHING consistently with each drop. If it is not, adjust the position of the plastic buret until it does.
12. Add approximately 2-3 mL of your dilute NaOH solution to the 10 mL graduated cylinder. Note down the exact volume in the cylinder (to one decimal place)
13. To calibrate the Drop Counter, tap “Volume” and select “Calibrate”
14. Tap “Calibrate Now” and then open the bottom tap on your plastic buret completely
15. After allowing about 2 mL of dilute NaOH to flow from the plastic buret into the 10 mL graduated cylinder, close the bottom tap
16. Note down the exact final volume in the 10 mL graduated cylinder and the number of drops (indicated on LabQuest 2)
17. Enter the “Precise Volume” in the designated box of your LabQuest 2 and tap “OK”
18. The Drop Counter is now calibrated. (you can pour the NaOH in the graduated cylinder back into your beaker).
19. Obtain a glass buret and label it “acid.” Attach the buret to the L bar using a double buret clamp
20. Use a 50 mL beaker to obtain approximately 25 mL of the standard acid. The standard acid is MONOPROTIC. NOTE its concentration exactly
21. Fill the glass buret with the standard acid using a funnel if needed. Verify that it is not leaking. Flow 1-2 mL of the acid through the tip of the buret, into a beaker, to ensure that there is no air trapped in the tap
22. Record the initial buret reading and then transfer approximately 10 mL of the standard acid into a clean 150 mL beaker. NOTE the EXACT final reading on the buret and calculate the EXACT volume of acid that was transferred. REMEMBER: The buret can be read to TWO DECIMAL PLACES
23. Add 2-3 drops of phenolphthalein and a magnetic stir bar to the beaker
24. Place the beaker of acid on a stir plate such that the solution from the plastic buret will drop into the beaker
25. Connect the pH probe in the CH1 port of the LabQuest 2
26. Rinse the tip of the pH probe with distilled water
27. Pass the pH probe through the hole in the Drop Counter.
28. Confirm that the pH probe is in the solution in the beaker and that the tip of the pH probe does NOT descend BEYOND THE 60 mL MARK ON THE BEAKER. You will need to add distilled water until the entire pH sensor is in the solution. * NOTE: To avoid the acid/base splashing out your beaker, try to minimize the distance between the plastic buret tip and the solution in your beaker
29. Refill your plastic buret to the 40 mL mark with dilute NaOH
30. Tap the graph icon and confirm that you are plotting pH as a function of volume
31. Start the run and then open the bottom tap completely to allow the base to flow into the

beaker at the rate of 1-2 drops/second

32. Note the volume of added base at which the colour of the acid solution changes
33. Stop the run after the pH has risen and then levelled off at about pH 11-12
34. Remove the pH probe and stirrer from the solution and rinse them with distilled water
35. Pour the contents of your beaker in the waste container and then rinse the beaker with distilled water
36. STORE the data in the Filing Cabinet. NOTE: Your data is NOT saved until you transfer it to your USB key!! It is only stored
37. Once again, fill the plastic buret to the 30 mL mark with dilute NaOH. Repeat the experiment at least once more. * You should have at performed at least three titrations with the standard acid. If you notice a substantial difference in the volumes at which you observe the colour change, you should perform an additional trial until you have two fairly reproducible volumes.
38. Empty the glass buret and rinse it with warm water and then distilled water, through the tip
39. Use a 50 mL beaker to obtain a 25 mL sample of the unknown acid assigned to you by your TA. The unknown acid is DIPROTIC. RECORD YOUR UNKNOWN NUMBER.
40. Fill the clean glass buret with your unknown acid and flow 1-2 mL of the acid through the tip, into a beaker, to ensure that there is no air trapped in the tap
41. Repeat steps 25-37 using the standardized NaOH solution – remember to top up the NaOH in your buret to 40 ml between each titration. * You should have at performed at least three titrations with your unknown acid. If you notice a substantial difference in the volumes at which you observe the colour change, you should do an additional trial until you have two fairly reproducible volumes.
42. SAVE your data, choosing the USB icon, giving your file a name, and then clicking “Save”
43. Remind your TA to save a copy of the data before you tap “File”, “New”.
44. Pour all solutions in the chemical waste container. Rinse the plastic and glassware used thoroughly with warm water and then with distilled water. Clean the counters of all paper towels before leaving
45. Use a wet paper towel to gently wipe the inner surface of the drop counter. Return all probes to your TA and turn off the LabQuest 2
46. Remember to get your raw data, written in PEN, signed by your TA and to attach this raw data AND your LABQUEST RAW DATA to your report in order to receive a grade!

Observations:

Table 1: Standardization of HCL (Concentration 0.100 mol/L) Using a Diluted NaOH Solution

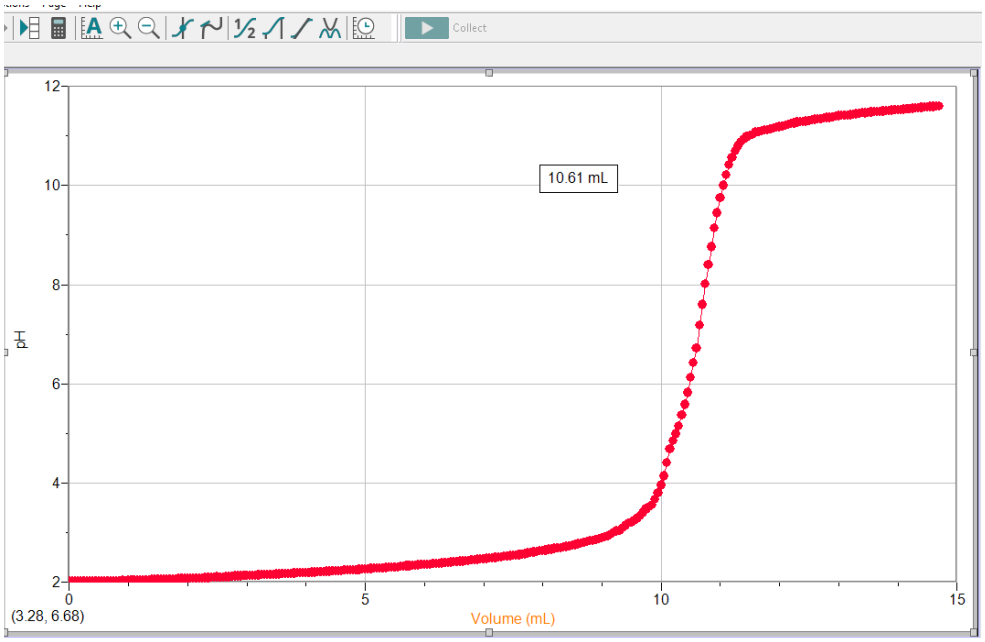
Volume (mL)	Trial 1	Trial 2	Trial 3

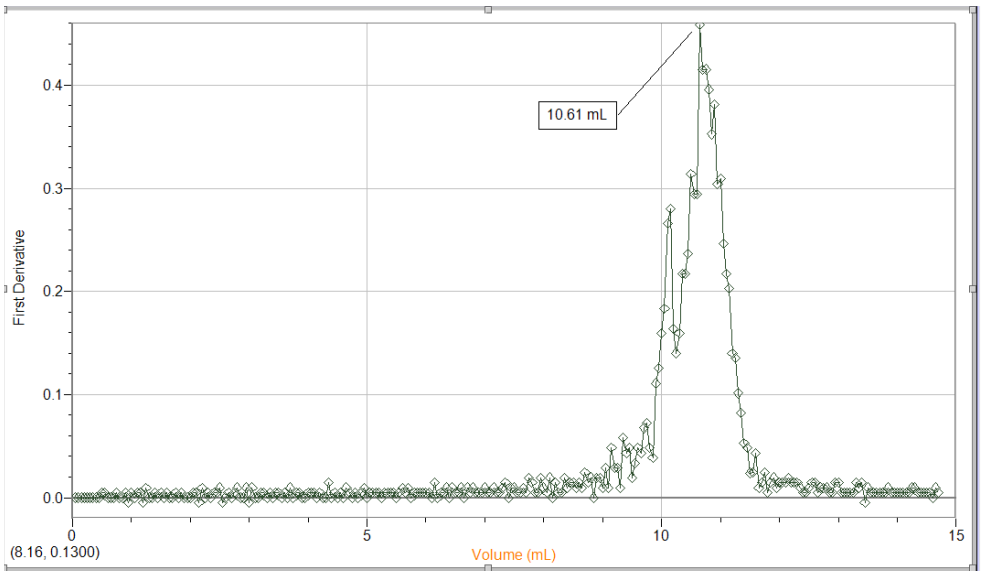
Volume of NaOH Solution t Equivalence Point	10.61	9.28	10.85

Table 2: Standardization of An Unknown Diprotic Acid Using a Diluted NaOH Solution

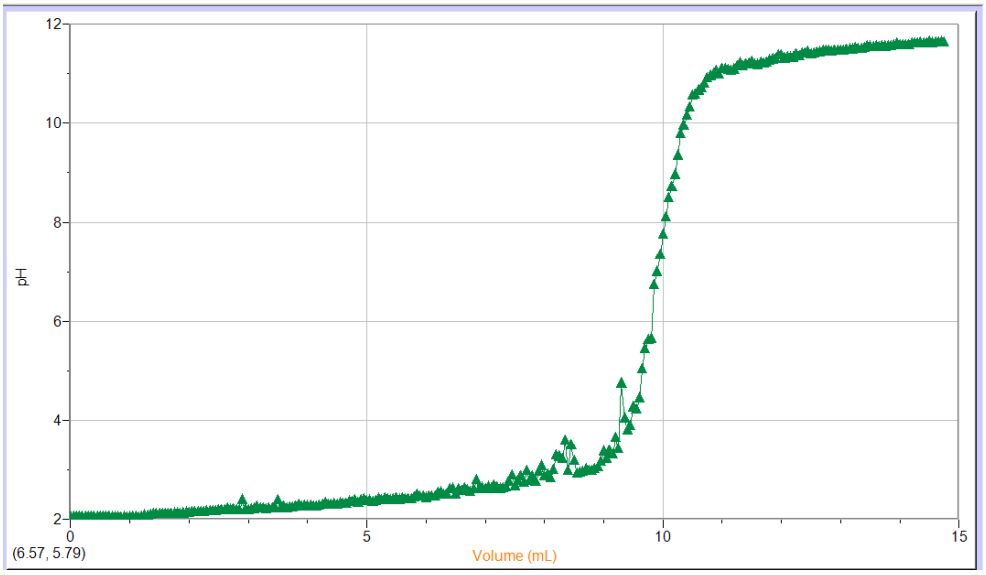
Volume (mL)	Trial 1	Trial 2	Trial 3
Volume of NaOH Solution at Equivalence Point	10.52	10.65	11.03

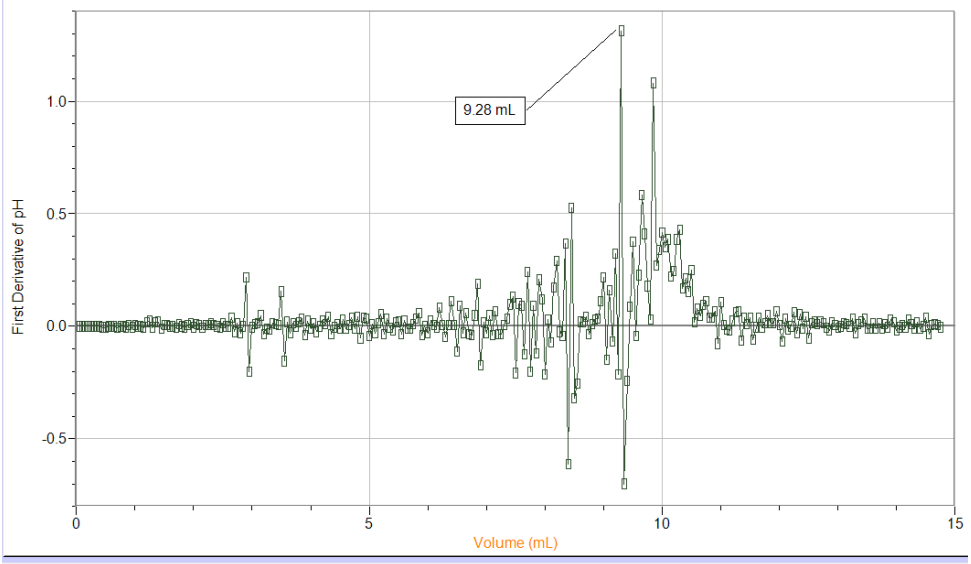
The Standardization of NaOH:
Trial 1



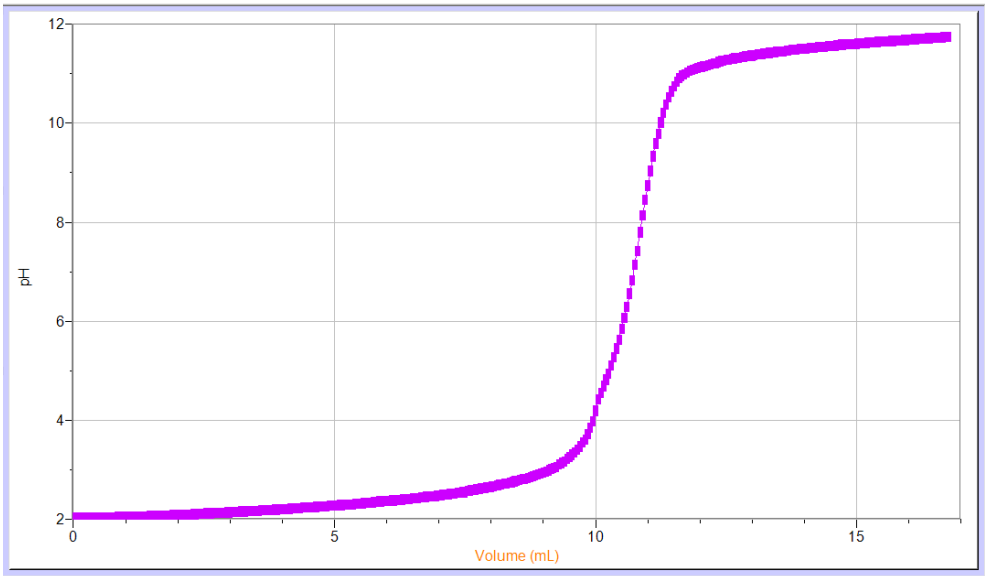

10.61 mL
First Derivative
0.4
0.3
0.2
0.1
0.0
0
5
10
15
(8.16, 0.1300)
Volume (mL)

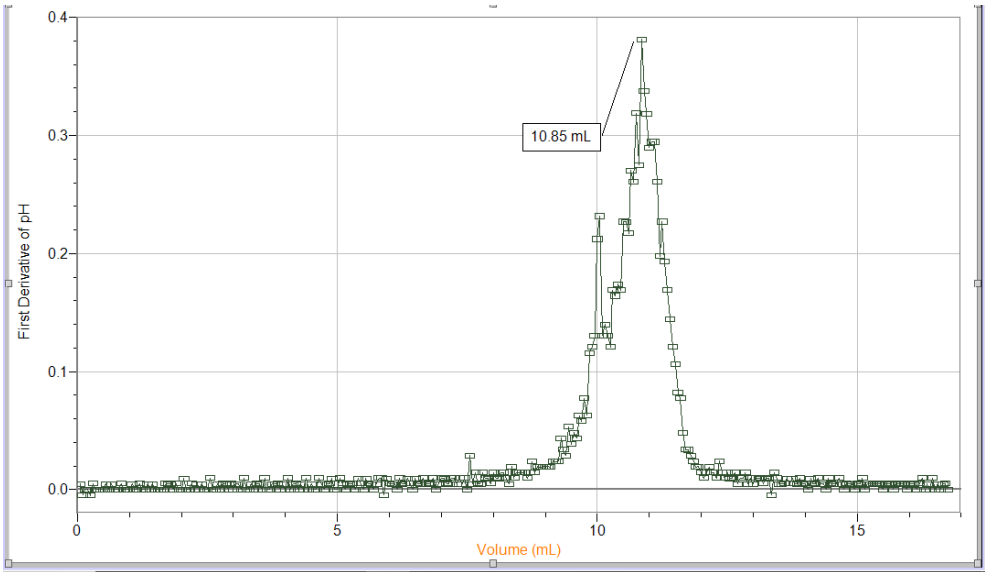
Trial 2


12
10
8
6
4
2
pH
0
5
10
15
(6.57, 5.79)
Volume (mL)

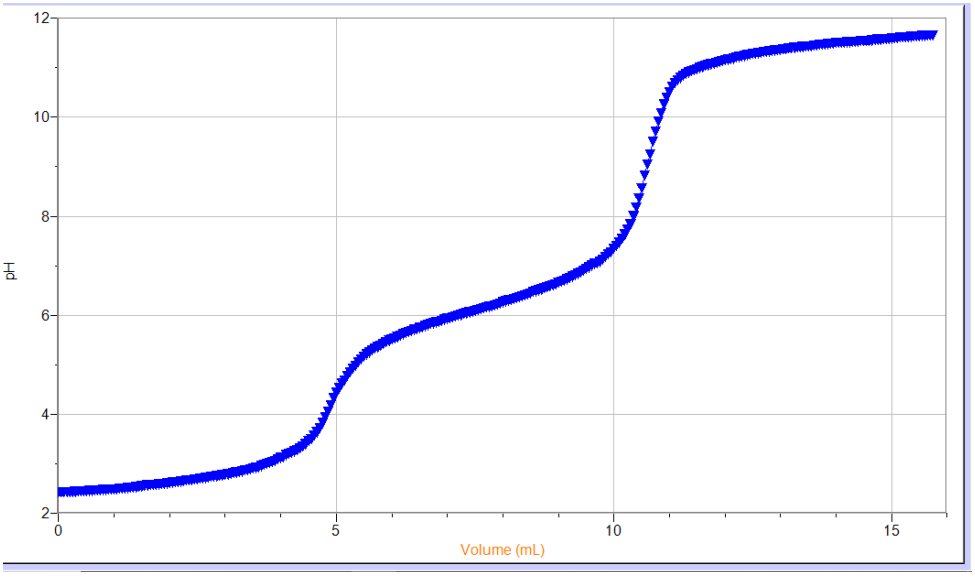

9.28 mL
First Derivative of pH
1.0
0.5
0.0
-0.5
0
5
10
15
Volume (mL)

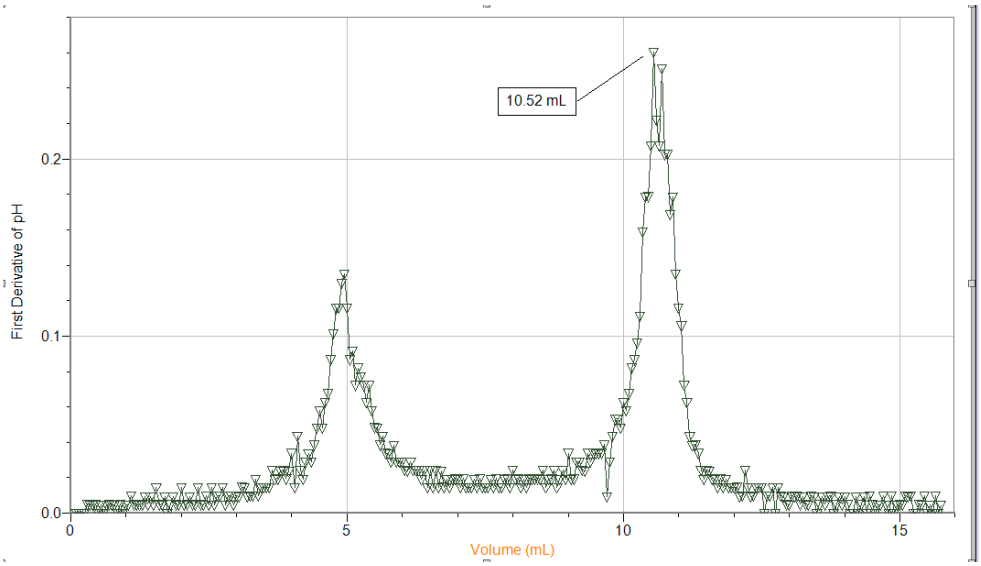
Trial 3


pH
12
10
8
6
4
2
0
5
10
15
Volume (mL)

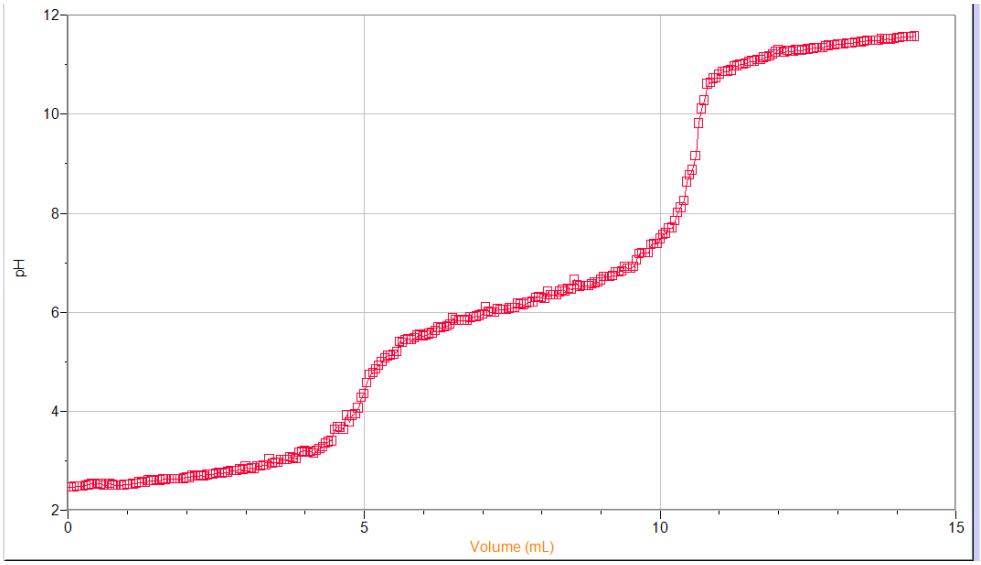


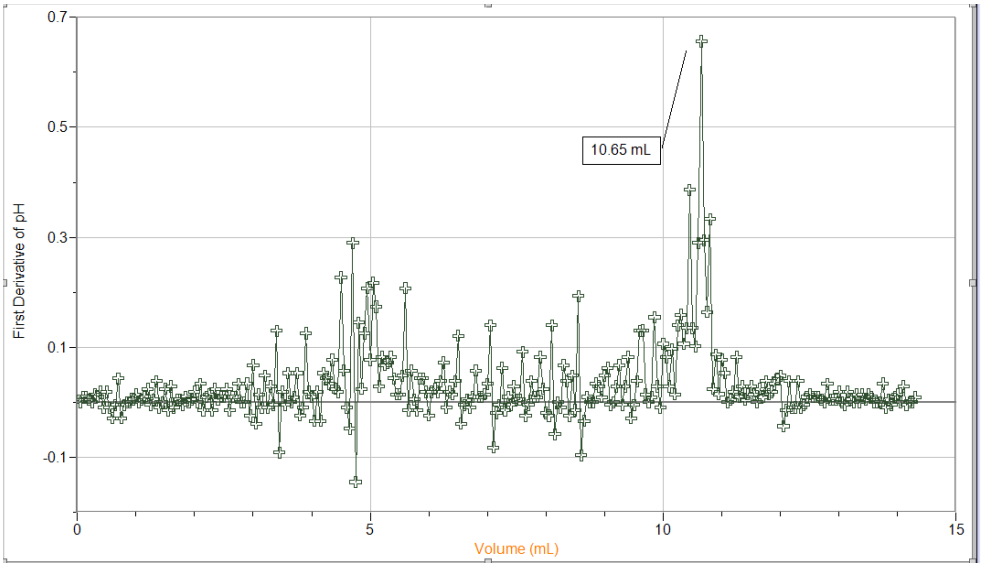
The Standardization of an Unknown Diprotic Acid:
Trial 1



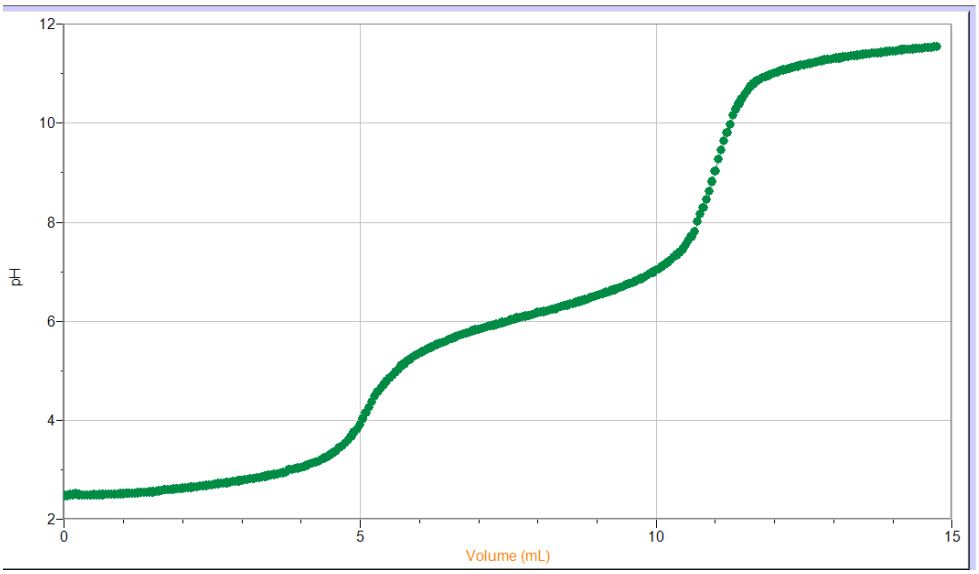

10.52 mL
First Derivative of pH
0.0
0.1
0.2
0
5
10
15
Volume (mL)

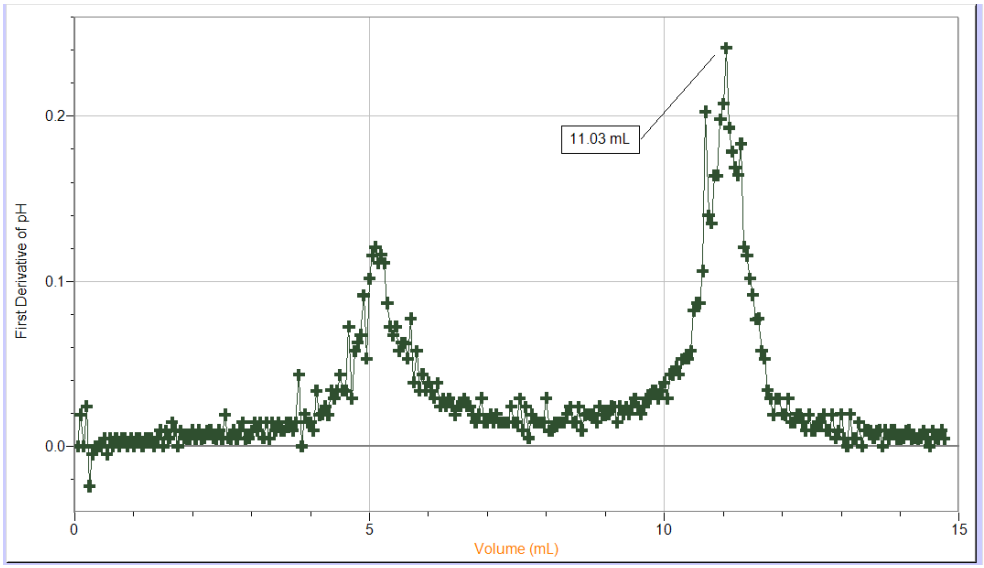
Trial 2


pH
2
4
6
8
10
12
0
5
10
15
Volume (mL)


10.65 mL
First Derivative of pH
-0.1
0.1
0.3
0.5
0.7
0
5
10
15
Volume (mL)

Trial 3





Analysis:

Standardization of the NaOH Solution: Sample Calculation for Trial 1

Initial Concentration of $HCl = 0.100\,mol/L$

Initial Volume of $HCl = 0.010\,L$

Final Volume of $HCl = 0.08\,L$

$C_1 V_1 = C_2 V_2$

$0.100\,mol/L \times 0.010\,L = C_2 \times 0.080\,L$

$C_2 = 0.0125\,mol/L$

$n = CV$

$n = 0.0125\,mol/L \times 0.08\,L$

$n = 1.00 \times 10^{-3}\,mols$

Mole Ratio= 1:1, therefore there is also $1.00 \times 10^{-3}\,mols$ of NaOH

$n = CV$

$C = n/V$

$C = \frac{1.00 \times 10^{-3} mols}{0.0106\, L}$

$C = 0.0943\, mols/L$

Table 3: Standardization of the NaOH Solution

	Trial 1	Trial 2	Trial 3
Concentration (mol/L)	0.0943	0.108	0.0922

Average Concentration of the NaOH Solution: 0.0982 mol/L

Standardization of the Unknown Acid Solution: Sample Calculation for Trial 1

$2\, NaOH + XH❑_2 \rightarrow NaX + 2\, H❑_2O$

Mole Ratio= 2:1

Volume of NaOH Solution= 0.01052 L

Concentration of NaOH Solution= 0.0982 mol/L

Volume of Unknown Diprotic Acid: 0.0800 L

$n = CV$

$n = 0.0982\, mol/L \times 0.01052\, L$

$n = 1.03 \times 10^{-3} mols\, NaOH$

¿$5.17 \times 10^{-4} mols\, XH❑_2$

$n = CV$

$C = \frac{n}{V}$

$C = \frac{5.17 \times 10^{-4} mols}{0.0800\, L}$

$C = 0.00646\, mols/L$

Table 4: Standardization of the Unknown Acid Solution

	Trial 1	Trial 2	Trial 3
Concentration (mol/L)	0.00646	0.00654	0.00677

Average Concentration of the Unknown Diprotic Acid Solution: 0.00659 mol/L

Average Concentration of the Unknown Diprotic Acid:

$C❑_1 V❑_1 = C❑_2 V❑_2$

$0.00659\, mol/L \times 0.0800\, L = C❑_2 \times 0.0100\, L$

$C❑_2 = 0.0520\, mol/L$

Therefore, the concentration of the unknown diprotic acid is 0.0520 moles per litre.

Conclusion:

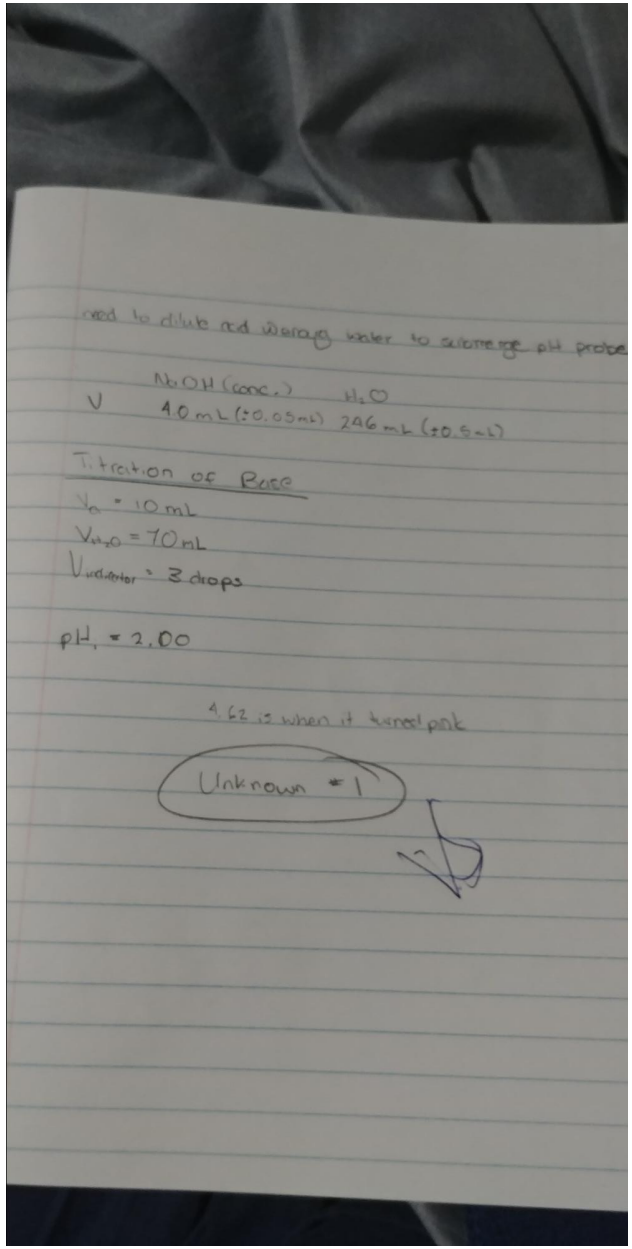
This experiment utilized a solution of HCl with a known concentration to find an accurate concentration for an NaOH solution. This concentration for the NaOH solution was then used to find the concentration of an unknown diprotic acid. The average concentration for the NaOH solution was found to be 0.0982 mol/L, while the concentration for the diprotic acid was found to be 0.0520 mol/L.

Although the concentration of the NaOH solution did not matter in terms of finding the concentration of the unknown diprotic acid (any solution of any concentration would have eventually yielded the same result), the solution was chosen to have 4 mL of the base added to 246 mL of water to ensure that the solution was not so strong that the results would be skewed, but strong enough that the experiment could be conducted in the time given.

There are, of course, errors inherent in an experiment like this, one being the occasional mishaps by the drop counter (technical errors and therefore unavoidable). Another would be the fact that the pH must be monitored in intervals, and therefore can only be so exact compared to a continuous measurement.

Appendices:

Run 2		Run 3		Run 4		Run 5		Run 6		Run 7	
Volume (mL)	pH	Volume (mL)	pH	Volume (mL)	pH	Volume (mL)	pH	Volume (mL)	pH	Volume (mL)	pH
0.000	2.01	0.000	2.04	0.000	2.03	0.000	2.45	0.000	2.46	0.000	2.47
0.050	2.01	0.050	2.04	0.050	2.04	0.050	2.45	0.050	2.46	0.050	2.47
0.100	2.01	0.100	2.04	0.100	2.03	0.100	2.45	0.100	2.47	0.100	2.49
0.150	2.01	0.150	2.04	0.150	2.03	0.150	2.45	0.150	2.48	0.150	2.49
0.200	2.01	0.200	2.04	0.200	2.03	0.200	2.45	0.200	2.49	0.200	2.52
0.250	2.01	0.250	2.04	0.250	2.03	0.250	2.45	0.250	2.49	0.250	2.49
0.300	2.01	0.300	2.04	0.300	2.03	0.300	2.45	0.300	2.50	0.300	2.49
0.350	2.01	0.350	2.04	0.350	2.03	0.350	2.46	0.350	2.51	0.350	2.49
0.400	2.01	0.400	2.04	0.400	2.03	0.400	2.46	0.400	2.53	0.400	2.49
0.450	2.01	0.450	2.04	0.450	2.03	0.450	2.47	0.450	2.52	0.450	2.49
0.500	2.01	0.500	2.03	0.500	2.03	0.500	2.47	0.500	2.54	0.500	2.49
0.550	2.02	0.550	2.03	0.550	2.04	0.550	2.47	0.550	2.53	0.550	2.49
0.600	2.02	0.600	2.03	0.600	2.04	0.600	2.47	0.600	2.50	0.600	2.49
0.650	2.02	0.650	2.03	0.650	2.04	0.650	2.48	0.650	2.50	0.650	2.49
0.700	2.02	0.700	2.03	0.700	2.04	0.700	2.48	0.700	2.54	0.700	2.50
0.750	2.02	0.750	2.03	0.750	2.04	0.750	2.49	0.750	2.51	0.750	2.50
0.800	2.02	0.800	2.03	0.800	2.04	0.800	2.49	0.800	2.51	0.800	2.50
0.850	2.02	0.850	2.02	0.850	2.05	0.850	2.49	0.850	2.51	0.850	2.51
0.900	2.03	0.900	2.03	0.900	2.05	0.900	2.50	0.900	2.51	0.900	2.51
0.950	2.02	0.950	2.02	0.950	2.05	0.950	2.50	0.950	2.51	0.950	2.51
1.000	2.03	1.000	2.03	1.000	2.05	1.000	2.50	1.000	2.53	1.000	2.51
1.050	2.03	1.050	2.03	1.050	2.05	1.050	2.51	1.050	2.53	1.050	2.52
1.100	2.03	1.100	2.03	1.100	2.05	1.100	2.52	1.100	2.53	1.100	2.52
1.150	2.04	1.150	2.02	1.150	2.05	1.150	2.52	1.150	2.54	1.150	2.52
1.200	2.03	1.200	2.04	1.200	2.06	1.200	2.53	1.200	2.56	1.200	2.53
1.250	2.04	1.250	2.07	1.250	2.06	1.250	2.53	1.250	2.56	1.250	2.53
1.300	2.04	1.300	2.06	1.300	2.06	1.300	2.54	1.300	2.56	1.300	2.54
1.350	2.04	1.350	2.08	1.350	2.06	1.350	2.54	1.350	2.60	1.350	2.54



need to dilute red wrapper water to submerge pH probe

	NaOH (conc.)	H_2O
V	4.0 mL (±0.05mL)	296 mL (±0.5mL)

Titration of Base

V_a = 10 mL

V_{H_2O} = 70 mL

$V_{indicator}$ = 3 drops

pH_i = 2.00

4.62 is when it turned pink

Unknown #1