Experiment 4: Titration

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Introduction

Acids and bases are defined by the Bronsted-Lowry theory, which states: a bases possess Hydrogen accepting properties and an acid posses Hydrogen donating properties. These two substances are used in many parts of life, such as blood pH management, body homeostasis, and neutralization reactions. Acids increase acidity, therefore decreasing pH and bases inversely increase pH. When they dissolve in water, strong acids dissociate to H^+ ions and bases dissociate to form OH^- ions.

Titrations using Acids and Bases involve an unknown concentration of one of these substances being determined by slowly titrating it against a standardized solution, usually an acid or base. The acid or base will neutralize the other, gradually increasing or decreasing the initial pH until it is close to 7 (Yang, Fan, Huang, Zhang, & Cao, 2011). The equivalence point is reached when the amount of acid is enough to react with the amount of base, or vice versa. Titration can have visual indicators that change the colour of the acid - base solution when a certain pH is reached. Therefore one should select an appropriate indicator for the desired pH. When the level of pH is reached and the colour changes, it's called an end point. If the volume at the equivalence point is the desired data, then one should choose an indicator that changes colour close to the equivalence point.

In this experiment, different concentrations of known and unknown acids were titrated against a standardized base. The equation to determine the concentrations of solutions is:

Concentration (*M*) = *amount of substance* (*mol*) / *volume of solution* (*L*)

This formula will give the amount of mols per litre of a substance in a solution. In this experiment, different unknown acids were titrated, and each one possessing a different number of removable protons when ionized. A monoprotic acid gives off 1 hydrogen ion in solution, diprotic gives 2 and triprotic gives 3 (Nguyen, Kao, & Kurtz, 2011).

Titration can be graphed by plotting the change in pH against the change in volume. Monoprotic acids can give off 1 proton during a reaction, therefore they have one equivalence point, whereas an acid with more than one removable proton can have as many equivalence points as they do removable protons. An equivalence point can be seen on a graph at the location of the steep curve. When reacting a polyprotic acid, more than one steep curve can be seen, indicating more than one equivalence point.

During an acid base reaction, the initial concentration of the acid and base separately will differ from the final concentrations of the 2 due to the substances combining their volumes which alters the pH. The final concentration of both can be calculated by this equation:

$$C_{acid} V_{acid} = C_{base} V_{base}$$

However, for diprotic acids, two H+ ions are released by each molecule, thus the stoichiometric ratios between the acid and base will be different, for example:

$$H_2SO_{4(aq)} + 2NaOH_{(aq)} \quad H_2O_{(l)} + Na_2SO_{4(aq)}$$

At the equivalence point: $n_{acid} = 2(n_{base})$ therefore:

$$c_{acid} V_{acid} = 2\ c_{base} V_{base}$$

The same is true for triprotic acids, however, the coefficient of the base will be three instead of two. Therefore, the general equation to find the concentration of the acid is:

$$(a) c_{acid} V_{acid} = (b) c_{base} V_{base}$$

Where (a) is the coefficient for the acid and (b) is that of the base in the chemical equation. The goal of this experiment was to determine the concentrations of an unknown acid sample and a juice sample, and determine the percent by mass of acid in the juice sample. This was done by titrating these unknown solutions with a standardized solution of NaOH.

Procedure

Procedure as outlined in the lab manual (OH HOW BITTER A THING IT IS…, R. Venkateswaran, 2017, p79-83)

Results and Observations

Table 1: Data for Part 1: formation of stock NaOH solution

Initial volume of NaOH	5mL
Volume of Water added	250mL
Final Volume of Stock Solution	255mL
Approximate concentration of NaOH and water	0.12M

Table 1 shows the initial concentration of NaOH was M, when mL of water was added, the final volume was mL. The solutions were both clear and colourless, before and after the addition of NaOH to the beaker. The NaOH was slightly thick and more dense than water. No change was observed when the two solutions were combined.

Table 2: Data for Part 2: standardization of stock solution of NaOH

Concentration of HCl	0.1M
Volume of HCl	10mL
Volume of NaOH solution added at end point	25.9mL
Average concentration of NaOH solution	0.0386M

Table 2 shows the initial concentration and volume of HCl acid. HCl was a clear colourless solution. 3 drops of phenolphthalein were added to the acidic solution before the titration. The colour at the end point was a bright pink.

Table 3: Data for Part 3: the titration of Unknown Acid #6

Volume of unknown acid	10mL
Volume of water added	75mL
Concentration of stock solution	0.0386M
Average Concentration of unknown acid #	0.086M

Table 3 shows the volume of the unknown acid and the NaOH solution. The unknown acid was a clear colourless solution that turned pink with the addition of 3 drops of phenolphthalein when the end point was reached.

Table 4: Data for Part 4: titration of Juice Sample #5

Volume of juice	10mL
Concentration of stock solution	0.0386M
Volume of NaOH solution added	25.28mL
Concentration of juice sample	0.0668 M
Density of juice	0.9993g/cm^3
Molar mass of acid in juice	192.14g/mol

Mass percent of acid in juice	1.2%

Table 4 shows the volume of unknown juice added, the concentration of the stock solution and juice sample. The juice was slightly tinted yellow. The solution turned pink when the end point was reach due to the addition of 3 drops of phenolphthalein.

<u>Graphs</u>

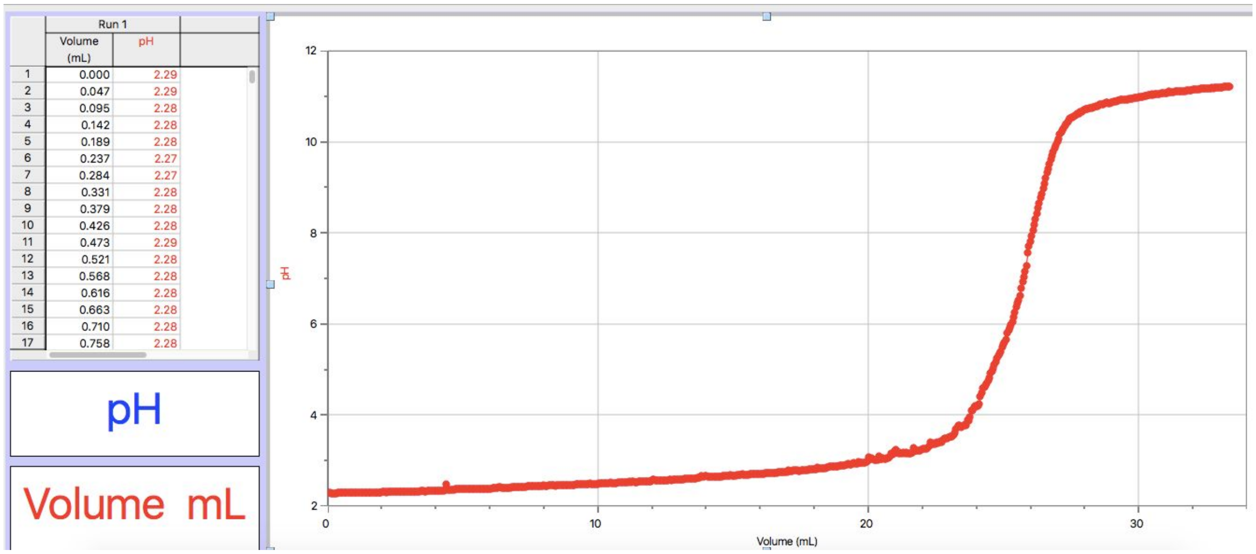


Figure 1: Titration of 10 mL 0.1mol/L $HCl_{(aq)}$ with 5 mL $NaOH_{(aq)}$

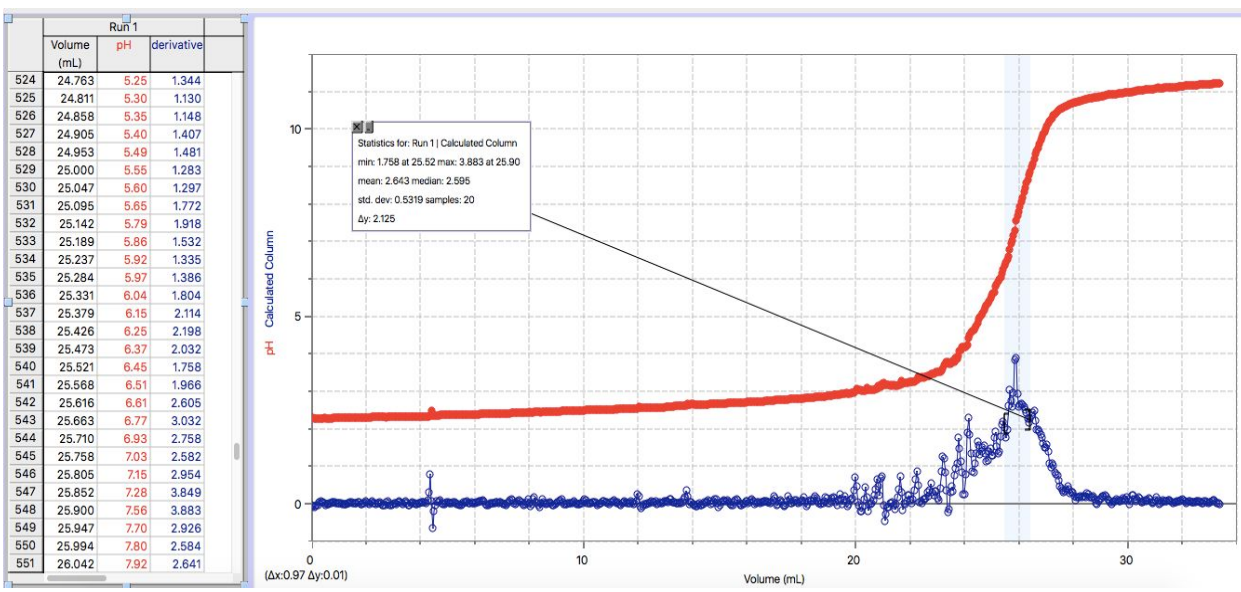
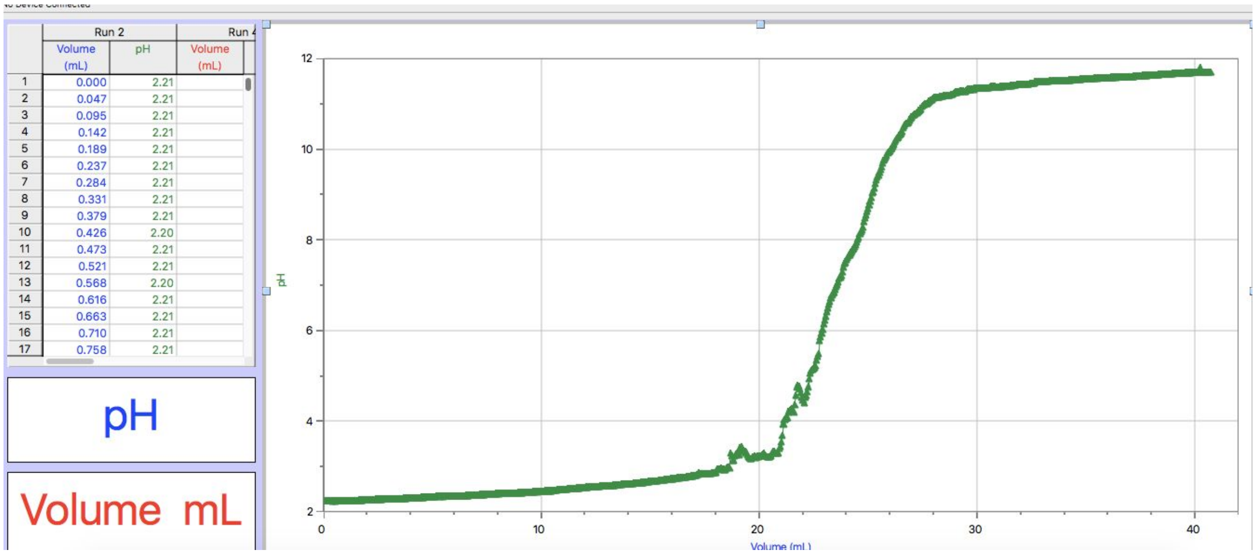
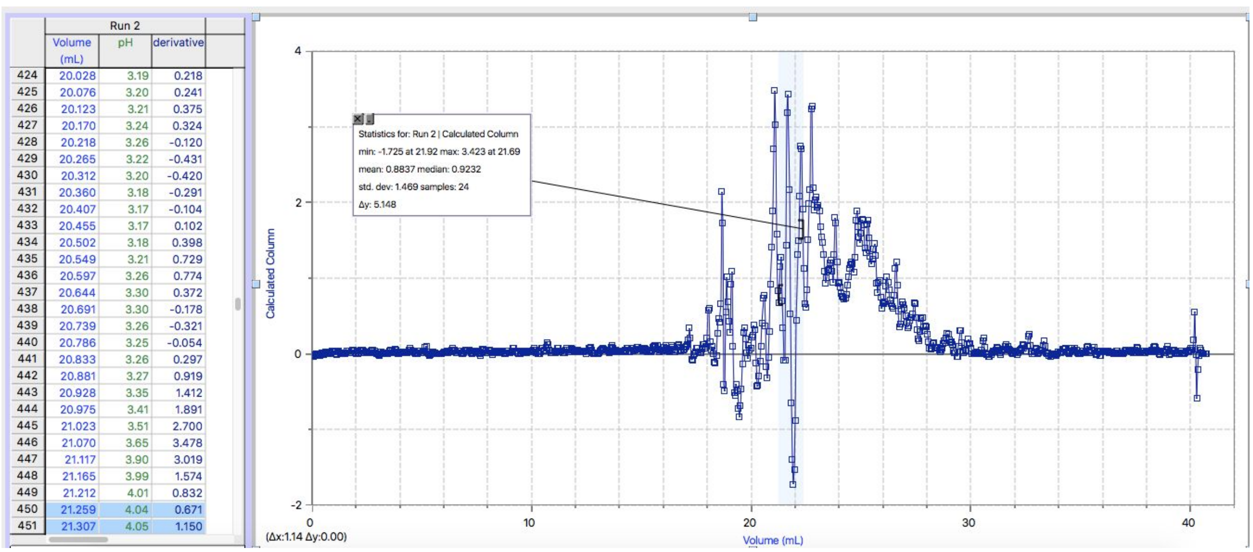


Figure 2: Derivative graph of pH vs volume of Run 1



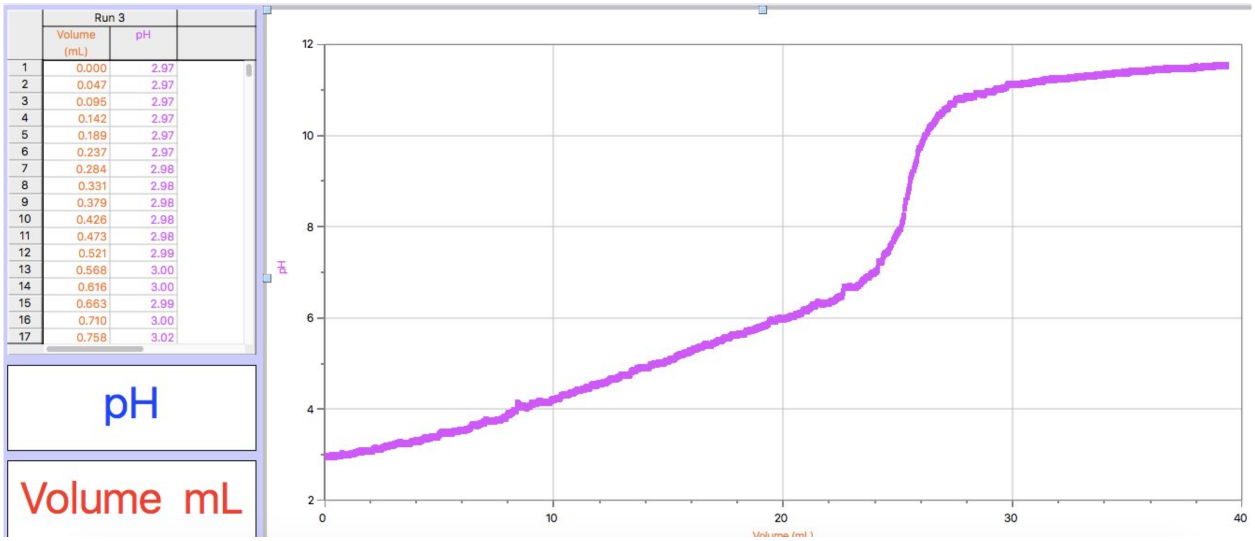
	Run 2 Volume (mL)	Run 2 pH	Run 4 Volume (mL)
1	0.000	2.21	
2	0.047	2.21	
3	0.095	2.21	
4	0.142	2.21	
5	0.189	2.21	
6	0.237	2.21	
7	0.284	2.21	
8	0.331	2.21	
9	0.379	2.21	
10	0.426	2.20	
11	0.473	2.21	
12	0.521	2.21	
13	0.568	2.20	
14	0.616	2.21	
15	0.663	2.21	
16	0.710	2.21	
17	0.758	2.21	

Figure 3: Titration of 10 mL of unknown acid with 5 mL $NaOH_{(aq)}$



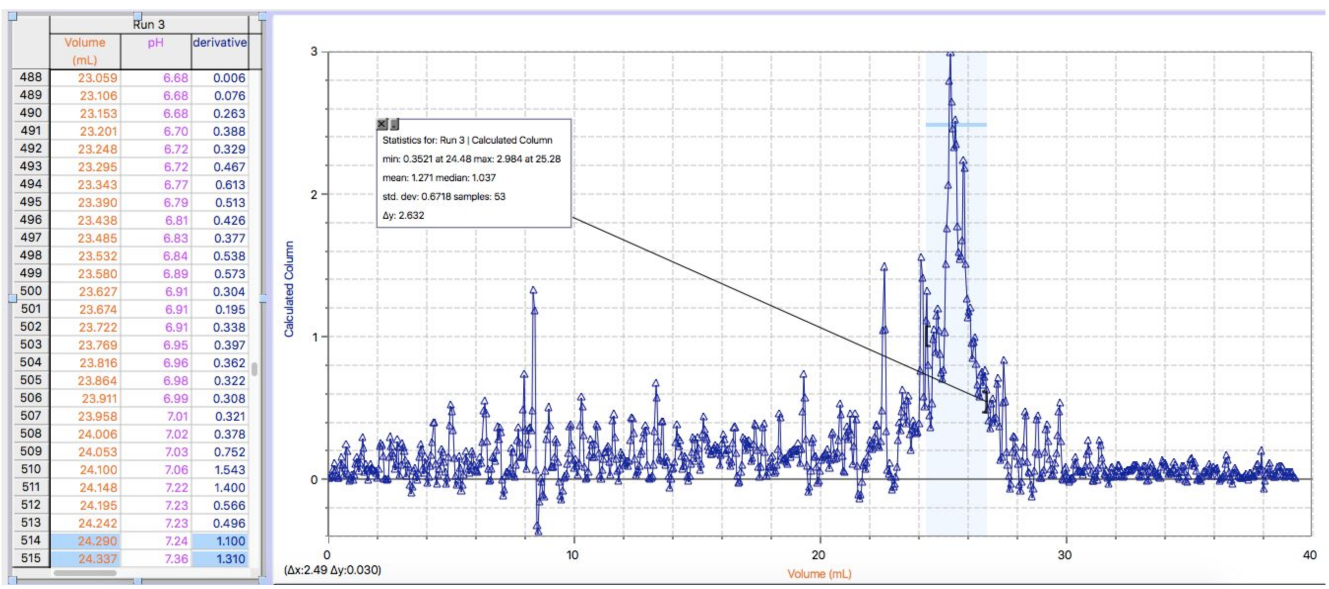
	Run 2 Volume (mL)	Run 2 pH	Run 2 derivative
424	20.028	3.19	0.218
425	20.076	3.20	0.241
426	20.123	3.21	0.375
427	20.170	3.24	0.324
428	20.218	3.26	-0.120
429	20.265	3.22	-0.431
430	20.312	3.20	-0.420
431	20.360	3.18	-0.291
432	20.407	3.17	-0.104
433	20.455	3.17	0.102
434	20.502	3.18	0.398
435	20.549	3.21	0.729
436	20.597	3.26	0.774
437	20.644	3.30	0.372
438	20.691	3.30	-0.178
439	20.739	3.26	-0.321
440	20.786	3.25	-0.054
441	20.833	3.26	0.297
442	20.881	3.27	0.919
443	20.928	3.35	1.412
444	20.975	3.41	1.891
445	21.023	3.51	2.700
446	21.070	3.65	3.478
447	21.117	3.90	3.019
448	21.165	3.99	1.574
449	21.212	4.01	0.832
450	21.259	4.04	0.671
451	21.307	4.05	1.150

Figure 4: Derivative graph of pH vs volume of Run 2



	Volume (mL)	pH
1	0.000	2.97
2	0.047	2.97
3	0.095	2.97
4	0.142	2.97
5	0.189	2.97
6	0.237	2.97
7	0.284	2.98
8	0.331	2.98
9	0.379	2.98
10	0.426	2.98
11	0.473	2.98
12	0.521	2.99
13	0.568	3.00
14	0.616	3.00
15	0.663	2.99
16	0.710	3.00
17	0.758	3.02

Figure 5: Titration of 10 mL of unknown juice with 5 mL $NaOH_{(aq)}$



	Volume (mL)	pH	derivative
488	23.059	6.68	0.006
489	23.106	6.68	0.076
490	23.153	6.68	0.263
491	23.201	6.70	0.388
492	23.248	6.72	0.329
493	23.295	6.72	0.467
494	23.343	6.77	0.613
495	23.390	6.79	0.513
496	23.438	6.81	0.426
497	23.485	6.83	0.377
498	23.532	6.84	0.538
499	23.580	6.89	0.573
500	23.627	6.91	0.304
501	23.674	6.91	0.195
502	23.722	6.91	0.338
503	23.769	6.95	0.397
504	23.816	6.96	0.362
505	23.864	6.98	0.322
506	23.911	6.99	0.308
507	23.958	7.01	0.321
508	24.006	7.02	0.378
509	24.053	7.03	0.752
510	24.100	7.06	1.543
511	24.148	7.22	1.400
512	24.195	7.23	0.566
513	24.242	7.23	0.496
514	24.290	7.24	1.100
515	24.337	7.36	1.310

Figure 6: Derivative graph of pH vs volume of Run 3

Calculations

Concentration of NaOH solution (Approximately)

$C_1V_1 = C_2V_2$

$C_2 = C_1V_1/V_2$

$C_2 = (6\ M)(\ 0.005L)/(\ 0.25L)$

$C_2 = 0.12\ M$

Concentration of NaOH solution (Exact) For Trial 1

From figure 2 - the amount needed to titrate the acid is 25.9 mL

$C_{NaOH}V_{NaOH} = C_{HCl}V_{HCl}$

$C_{NaOH} = C_{HCl}V_{HCl}/V_{NaOH}$

$C_{NaOH} = (\ 0.1M)(\ 0.01L)/(\ 0.0259L)$

$C_{NaOH} = 0.0386M$

Average concentration of stock solution of NaOH

$C_{av} = C_{T1}+C_{T2}\ /\ 2$

$C_{av} = (\ 0.0386M)+(\ 0.12M)\ /2$

$C_{av} = 0.0793M$

Concentration of unknown acid #1 . Unknown Acid #1 is diprotic: a = 2.

From Figure 4, exact volume of NaOH required to titrate unknown acid #1 = 21.69 mL

$(b)C_{NaOH}V_{NaOH} = (a)C_{Acid}V_{Acid}$

$C_{Acid} = (b/a)C_{NaOH}V_{NaOH}/V_{Acid}$

$C_{Acid} = (1/2)(0.0793\ M)(21.69\ mL)/(0.01mL)$

$C_{Acid} = 0.086M$

Concentration of acid in juice sample #2 (calculation for Trial 1). Juice #2 is triprotic: a = 3

From Figure 6, the exact volume of NaOH required to titrate juice sample #2 = 25.28 mL

$(b)C_{NaOH}V_{NaOH} = (a)C_{Acid}V_{Acid}$

$C_{Acid} = (b)C_{NaOH}V_{NaOH}/V_{Acid}\ (a)$

$C_{Acid} = (1/3)(0.0793\ M)(25.28\ mL)/(0.01mL)$

$C_{Acid} = 0.0668\ M$

Mass percentage of acid in juice. Acid in juice is citric acid ($H_3C_6H_5O_7$) with molar mass (MM) 192.14g/mol

Mass percent of acid in juice = 0.451 %

$= [(CAcid)(MMacid)\ /\ (densityjuice)*1000mL/\ L] * 100\%$

$= [(0.0668M)(192.14g/mol)\ /\ (0.9993g/cm^3)*1000mL/\ L] * 100\%$

=1.2%

Discussion

After the results of this experiment were calculated, it was determined that the concentration of the unknown acid was 0.0668M and the mass by percent of the acid in the juice is 1.2%. As the actual values are not available, it is impossible to know what the percent error actually is. However, there are a few possible sources of error for this lab. One of the errors is the repeated use of the same beaker. The beaker was washed after every trial, however there are still traces of acid/base in the beaker, which could cause the substance to reach its equivalence point faster/slower than it otherwise would. Another source of potential error is the drop counter accurately counting drops, the drop counter could have missed any number of drops which would lead to an incorrect amount of NaOH being used in the titration. All of these potential errors will cause inaccurate titration curves as well as inaccurate calculated equivalence points; the NaOH required will be overstated, and thus, the calculated concentration of the acids will be overstated as well.

The volume of NaOH used in the beginning does not matter as long as there are enough moles of NaOH to react completely with the moles of acid during the titration. The final concentration of the stock solution is dependent on the initial volume of concentrated NaOH, and the amount of water added to it. Changing the initial volume of concentrated NaOH will only affect the volume of stock solution required to reach the endpoint of the titration, however, the molar amount of NaOH required will always be constant. The titrant must also be in excess when performing a titration. As long as the moles of NaOH are enough to react with the acid, the volume doesn't matter.

The concentration of NaOH was calculated first because it was diluted with water in the second step, meaning the concentration would change. We need an accurate measurement of concentration because the rest of the calculations were done using that concentration therefore the more accurate it is, the better. This is why the base solution was titrated with the known acid, to have a better measurement of its concentration.

Logger Pro values would be more accurate in locating the volumes at equivalence point compared to an observed value seen during a colour change. This is due to potential experimental errors that could have occurred during the titration process. However the values observed were very similar to those calculate by Logger Pro, they only differed by decimal amounts.

There is a factor of 1000 in the equation for mass percent of acid in the juice because it's conversion factor to show the density in terms of grams per litre. The given density has the units g/cm3 , which is equivalent to g/mL. Thus, multiplying the density by a factor of 1000 mL/L is required to convert this to g/L.

The unknown juice sample contained citric acid which was a triprotic acid, so it's titration involved 3 steps to remove the 3 protons (Cabot, Fuguet, Ràfols, & Rosés, 2013). In the first step, the acid loses it's first proton

$$H_3C_6H_5O_{7(aq)} + NaOH_{(aq)} \rightleftharpoons NaH_2C_6H_5O_{7(aq)} + H_2O_{(l)}$$

Before any amount of base is added, the acid will still dissociate in water, however there will be more reactants than products because citric acid is a weak acid. After the base NaOH is added, the reaction will start to produce products, and at the midpoint of the first equivalence point, the concentration of acid will be equal to that of the deprotonated acid and the sodium ion:

$$[H_3C_6H_5O_{7(aq)}] = [H_2\,C_6\,H_5\,O_{7\,(aq)}{}^-] = [Na+]$$

At the first equivalence point, the citric acid has released 1 proton, which means the ratio of $[H_3C_6H_5O_7]$ to $[H_2C_6H_5O_{7}-]$ will be 0:1. The molar ratio of $H_2C_6H_5O_{7}-$ to Na^+ is 1:1. The second step of the reaction shows the acid neutralizing the base:

$$NaH_2C_6H_5O_{7(aq)} + NaOH_{(aq)} \rightleftharpoons Na_2HC_6H_5O_{7(aq)} + H_2O_{(l)}$$

At the midway point of this neutralization, the concentration of the acid that now only contains 2 protons is equal to the acid that contains 1:

$$[H_2\,C_6\,H_5\,O_{7(aq)}\,-] = [HC_6\,H_5\,O_7{}^{2-}{}_{(aq)}]$$

At the second equivalence point, the acid containing 1 proton is no longer in the solution, now the ratio of $HC_6H_5O_{7}2-$ to Na+ is now 1:2. The third step in the neutralization of citric acid is:

$$Na_2HC_6H_5O_{7(aq)} + NaOH_{(aq)} \rightleftharpoons Na_3C_6H_5O_{7(aq)} + H_2O_{(l)}$$

The pattern for the third midpoint of for the equivalence points continues, this is the midway point to the third equivalence point:

$$[HC_6\,H_5\,O_{7}{}^{-}] = [C_6\,H_5\,O_7{}^{3-}]$$

At the third equivalence point, there is no more acidic substance, and the ratio of $C_6H_5O_7^{3-}$ to Na+ is 1:3.

Conclusion

In this lab, the concentration of an unknown acid and the concentration and weight percent of an acid in an unknown juice sample was determined by titrating an unknown sample with a known sample. The concentration of the unknown acid was 0.086M for trial 1 and the concentration of the acid in an unknown juice sample was found to be 0.0668 M for trial 1 with a weight percent of 1.2%.

References

Cabot, J. M., Fuguet, E., Ràfols, C., & Rosés, M. (2013). Determination of acidity constants by the capillary electrophoresis internal standard method. IV. Polyprotic compounds. *Journal of Chromatography A*, *1279*, 108–116.

Nguyen, M. K., Kao, L., & Kurtz, I. (2011). Defining the buffering process by a triprotic acid without relying on Stewart-electroneutrality considerations. *Theor Biol Med Model*, *8*(29), 11. Retrieved from http://www.ncbi.nlm.nih.gov/pubmed/21849064

Yang, Q., Fan, L. Y., Huang, S. S., Zhang, W., & Cao, C. X. (2011). Equivalence-point electromigration acid-base titration via moving neutralization boundary electrophoresis. *Electrophoresis*, *32*(9), 1015–1024.

Ex. 4

250mL in beaker (water) pH = 4.29
5mL NaOH 6mol/L in cylinder

0.1m/L HCl - monoprotic 25mL
10mL HCl in beaker

(1) 3 drops 0.1% phenolphthalein
~~+100~~ 125mL water
pH = 2.30

turned pink @ 25mL NaOH added

(2) 10mL of an unknown acid #1 → diphrotic
add 75mL water
pH = 2.22
turned pink at 29 mL
3 drops phenolphthalein
after 29 mL pH = 9

*calculate concentration of unknown acid

(3) 10mL of unknown juice #2 triprotic
ρ = 0.9993 g/cm³
95mL water
3 drops phenolphthalein
pH = 2.97
after 30mL, pH = 11.19
final pH = 11.55