

Experiment 4: Acid Base Titrations

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A substance can be determined to be either acidic or basic based on the proton concentration of the substance in question. An acid is a substance with a greater concentration of protons than water, and therefore has a pH below seven. Acids have many defining properties—the primary one being that they will always donate their hydrogen ions to the base in the solution. However, the donation of a proton is only one defining property, and is described in the Brønsted-Lowry definition of an acid. The Brønsted-Lowry definition however, ignores the acid's other important property; the ability to act as an electron acceptor, as described in the Lowry definition of an acid. Thus, an acid is both a proton donor, and an electron acceptor. Likewise, any base will act to neutralize an acid. Therefore, a base is both a proton acceptor and an electron donor.

Titration is an integral part of both a student's and a professional's life. Titrations are very commonly used by students to determine the concentration of a substance, and this method is used in the development and control of concentration of new drugs. The substance with the unknown concentration is also known as an analyte, and the solution it is being added to is called the reagent. A titration is performed by placing the analyte in a beaker under a burette and adding an indicator. After the analyte is placed, the burette can be opened to drip the reagent slowly into the beaker. When a colour change occurs, it means that the acid has fully neutralized the base, or vice versa. The point at which neutralization occurs is known as an endpoint.

The endpoint of any reaction is when the reaction has gone to completion, and all of the reactants are finished interacting with each other. For a titration, the endpoint of the experiment is indicated by the colour change that is caused by adding an indicator such as phenolphthalein. A titration also has a factor known as an equivalence point, which is similar in concept to an endpoint. The equivalence point in a titration is when the number of moles of the acid is equal to the number of moles of the base—the solution is equal parts acid and base. When the colour change occurs in a titration, it is important to close the burette immediately, to keep the reaction from continuing past the endpoint and over-saturating the solution with an acid or a base.

The concept of an equivalence point is what allows the unknown concentration of the analyte to be found. At the equivalence point, the acid and the base have an equal number of

moles. One way to solve the concentration of the unknown analyte is using the number of moles. The number of moles of the reagent and the analyte is equal to the molarity of the reagent multiplied by the volume of the reagent. This number can then be multiplied by the volume of the analyte to find the concentration of the analyte. This method can be simplified to use just the concentration and volume of the analyte and reagent, since the number of moles is equal. The simplified method is demonstrated in the equation below:

$$C_{\text{acid}}V_{\text{acid}} = C_{\text{base}}V_{\text{base}} \quad [1]$$

A solution often must be standardized before the concentration of the unknown acid can be determined. Standardization is used to find the concentration of the solution that will then be used when finding the concentration of the unknown acid. The reason NaOH has to be standardized is because this compound absorbs water when exposed to air, which changes the concentration of the solution. NaOH also absorbs carbon dioxide when it comes into contact with air, which will produce sodium carbonate in the solution and change the overall concentration of NaOH. Standardizing the NaOH allows the NaOH to maintain a stable concentration, which can be calculated to use in other processes.

Procedure: As described in the lab manual “Oh How Bitter a Thing it Is...” Acid Base Titrations, Rashmi Venkateswaran

Observations:

- 5mL (from bottom of meniscus) of 6M of NaOH was measured
- 250mL (from bottom of meniscus) was added to a beaker
- Addition of NaOH initially created an oily texture in the mixture, which dissipated after approximately 30 seconds

Standardizing the Dilute NaOH Solution

- This portion of the experiment was completed using a monoprotic acid with a concentration of 0.100M
- For each trial, distilled water was added until the contents of the beaker reached 100mL (approximately 90mL of distilled water)
- 3 drops of phenolphthalein was added for each trial

Table 1. Data and Results in the Standardization of the Dilute NaOH Solution

Trial	Initial Burette Volume (mL)	Final Burette Volume (mL)	Exact Volume Transferred (mL)	Amount of NaOH added for colour change to occur (mL) (visual value)	Amount of NaOH added for colour change to occur (mL) (LabQuest value)	Stabilized pH	Final volume (mL)
1	2.00	12.00	10.00	10.00	23.60	11.16	11.00
2	12.00	22.10	10.10	10.00	24.00	11.44	13.00
3	11.00	21.00	10.00	10.00	25.60	11.54	15.00
4	11.00	21.00	10.00	9.00	25.20	11.33	12.00

Determining Concentration of Unknown Acid

- For each trial, distilled water was added until the contents of the beaker reached 100mL (approximately 90mL of distilled water)
- Experiment was completed with unknown acid number one
- 3 drops of phenolphthalein was added for each trial

Table 2. Data and Results in the Determination of the Concentration of the Unknown Acid

Trial	Initial Burette Volume (mL)	Final Burette Volume (mL)	Exact Volume Transferred (mL)	Amount of NaOH added for colour change to occur (mL) (visual value)	Amount of NaOH added for colour change to occur (mL) (LabQuest value)	Stabilized pH	Final volume (mL)
1	2.00	12.00	10.00	10.00	25.87	11.46	13.00
2	12.00	22.00	10.00	10.00	25.73	11.68	16.00
3	11.00	21.00	10.00	10.00	27.20	11.66	14.00

4	10.00	20.00	10.00	10.00	26.67	11.34	16.00
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Calculations:

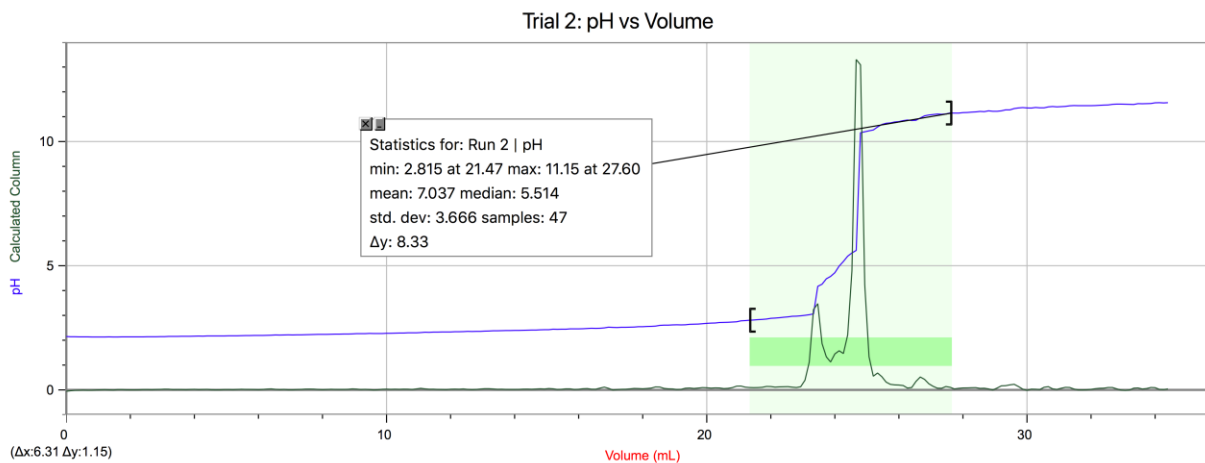
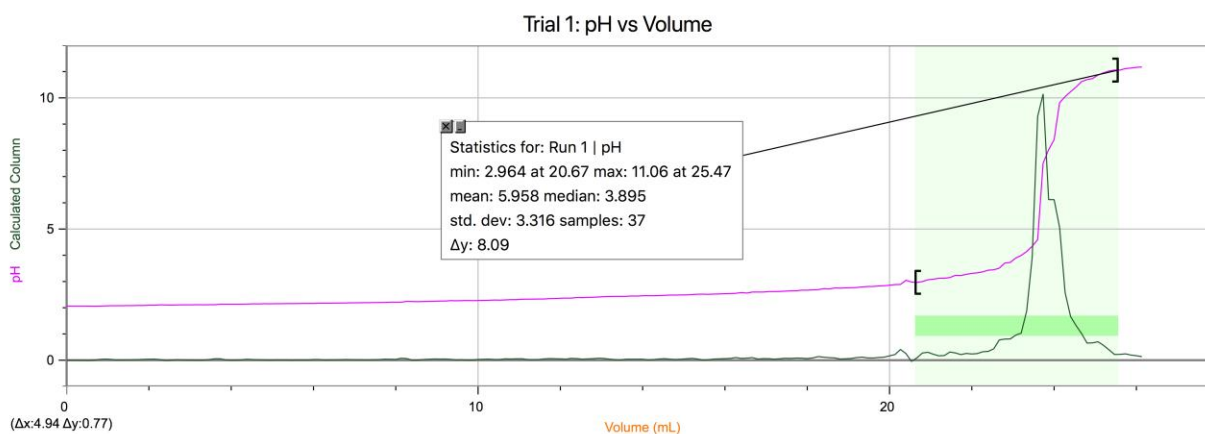
- Determining the concentration of the dilute NaOH solution:

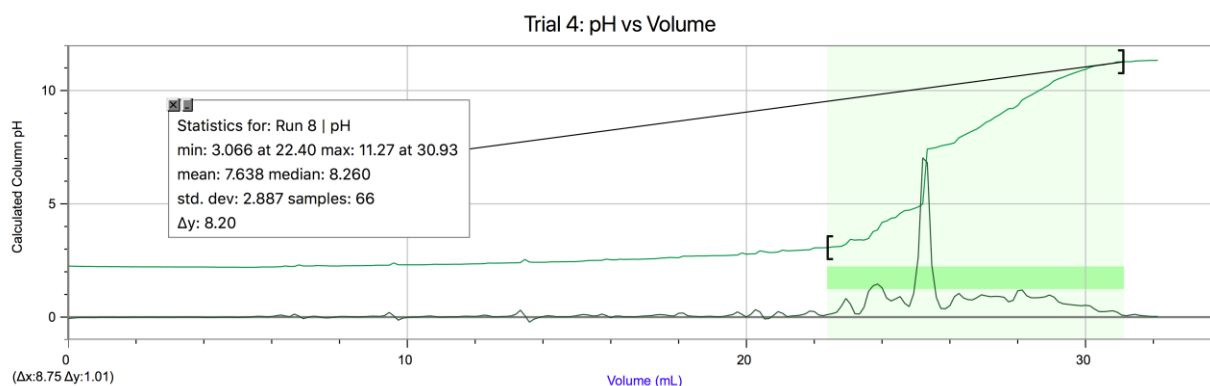
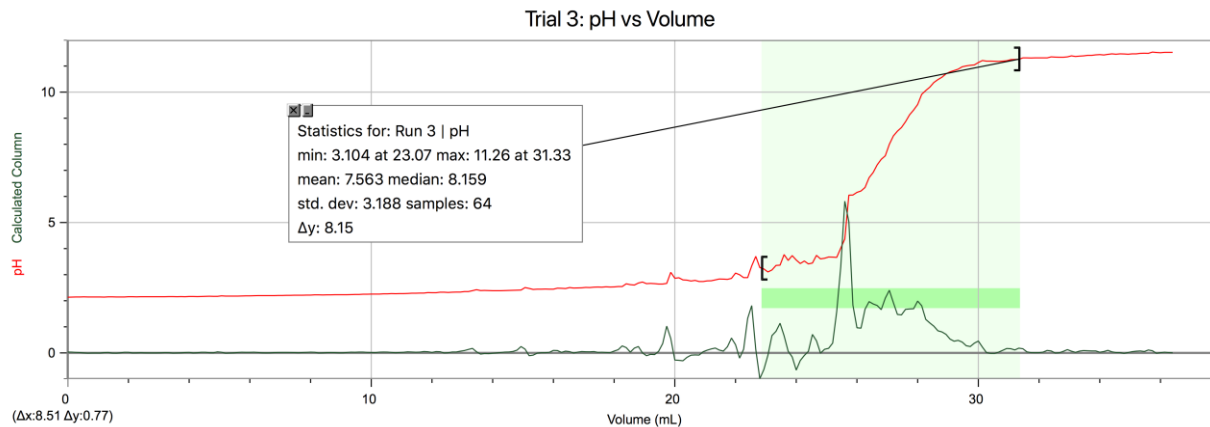
V_1 = initial volume of NaOH = 5mL, V_2 = initial volume of NaOH plus volume of distilled water added = 255mL, C_1 = initial concentration of NaOH solution, C_2 = concentration of dilute NaOH

$$C_2 = \frac{C_1 \times V_1}{V_2} = \frac{(6M)(0.005L)}{0.255L} = 0.1176M = 0.12M$$

- Graphs

Standardization of Dilute Solution:





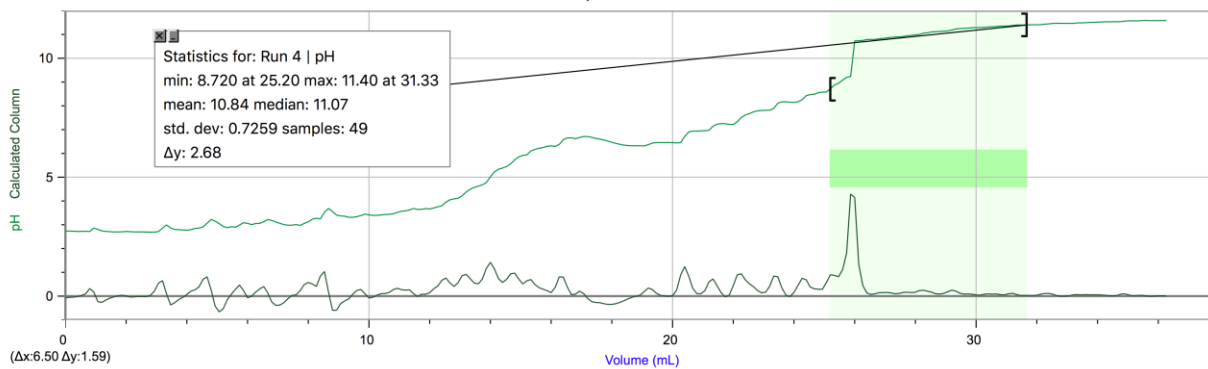
If the equilibrium point from the LabQuest data was used, the dilute base would have a concentration of:

$$\text{Trial 1: } C_{\text{base}} = \frac{2C_{\text{acid}}V_{\text{acid}}}{V_{\text{base}}} = \frac{2(0.100M)(0.01L)}{0.02547L} = 0.079M$$

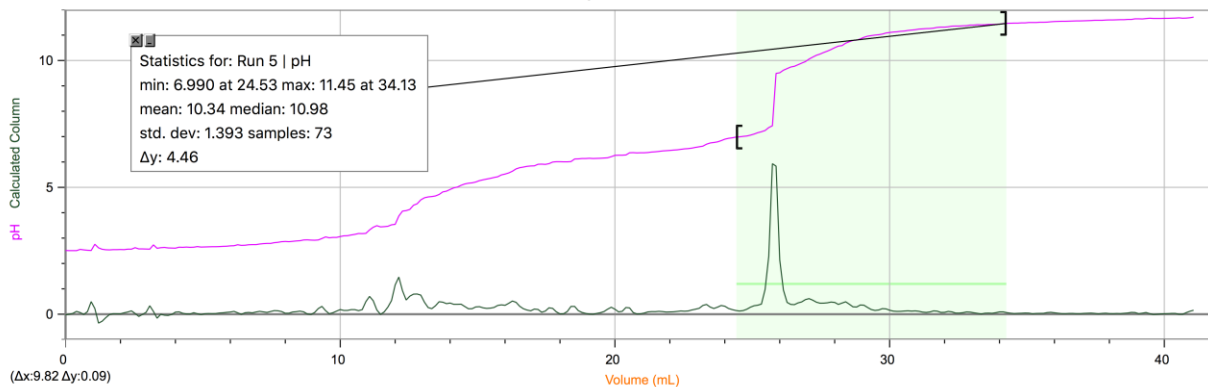
This value is significantly different from the value calculated in previous steps, since different values were used for the volume of the base. The LabQuest and drip count were incorrectly calibrated, so the visual value was used instead for calculations. The visual value will be less accurate than the value that would have been calculated by LabQuest, had it been calibrated correctly. Despite the technical error, the trends displayed on the graph are correct. The graphs demonstrate that the pH will exponentially increase as the volume reaches a certain point. The point at which the pH begins to significantly increase is near the endpoint. The derivative of the pH function shows when the equilibrium point occurred, because at this point the pH leveled off. The pH levelling off is an indication that the reaction is not continuing and the acid/base has been neutralized.

Determining the concentration of the unknown acid:

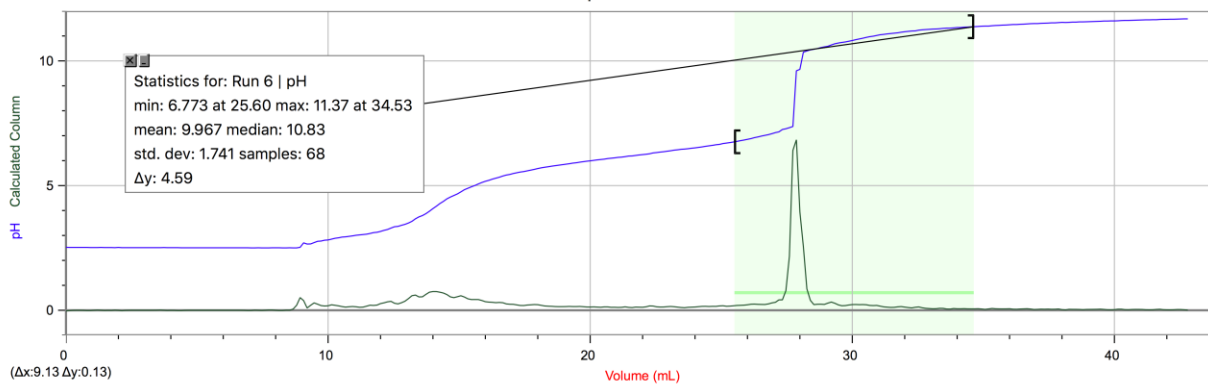
Trial 1: pH vs Volume

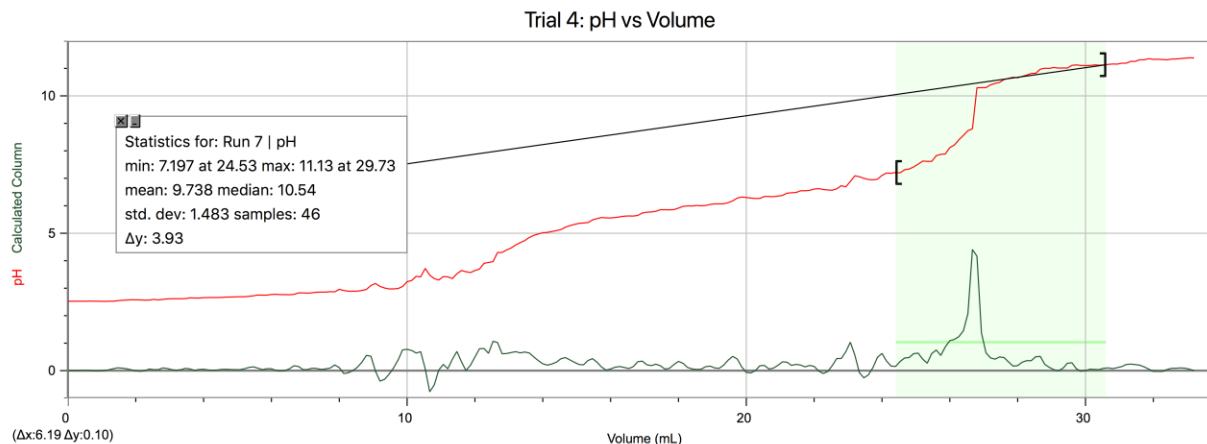


Trial 2: pH vs Volume



Trial 3: pH vs Volume





If the equilibrium point from the LabQuest data was used, the unknown acid would have a concentration of:

$$\text{Trial 1: } C_{\text{acid}} = \frac{C_{\text{base}}V_{\text{base}}}{2V_{\text{acid}}} = \frac{(0.079M)(0.031L)}{2(0.01L)} = 0.12M$$

This value is double the value that was calculated using visual methods, which shows how inaccurate the drip count calibration was. Regardless, like the first part of the experiment, the trend on the graph is accurate, even if the numbers are not. The trends on the graph for the second part of the experiment also show that as the pH plateaus, the maximum value of the derivative is reached, which is also the equivalence point.

3. Determining the concentration of the unknown acid:

V_{acid} = volume of acid added to NaOH solution, V_{base} = volume of base added when colour change occurred (the visual value is used for the calculation instead of the LabQuest value due to calibration issues), C_{base} = the concentration of the base in the dilute NaOH solution

$$C_{\text{acid}} = \frac{C_{\text{base}}V_{\text{base}}}{2V_{\text{acid}}} = \frac{(0.12M)(0.01L)}{2(0.01L)} = 0.06M$$

- The same values for the volume of the acid and base occurred for all trials, so the concentration of the unknown acid remained consistent

Points to Ponder:

1. The volume of concentrated NaOH used in the beginning does not matter, however recording the exact value used is very important. For example, one student could use 4.80mL and another could use 5.0mL, and their experiments should not yield different results, provided they correctly recorded the initial volume.

2. The concentration of the NaOH is determined right before it is used because the new, dilute concentration is what is required for the determination of the unknown acid. As well, the concentration of the NaOH needs to be known at the point just before standardization, because this concentration will remain constant after standardization.
3. The volumes at which the colour change occurred (as an approximate number observed on LabQuest) was less than the value demonstrated on the derivative graph on the equivalence point. This error occurred during both the standardization of the base and the determination of the unknown acid. The difference in volume is likely due to human error, because the volume that was written down was when the colour change initially occurred, which may have been just before the true equivalence point.
4. The error encountered that was associated with the calibration error on the LabQuest decreased the concentration of the unknown acid because the volume was incorrectly recorded to be more than double the value it actually was.
5.
 - a) At 0mL of added base there would only be the unknown acid and the phenolphthalein
 - b) At midway to the first equivalence point (which is at the top of the first slight curve of the pH derivative function), half of the unknown acid's protons will have dissociated into its conjugate base. The species present are the unknown acid (H_2X), NaOH, water and NaHX. Species from both the product and reactant sides are present as the reaction has not gone to completion.
 - c) At the first equivalence point, all of the acid's protons will have dissociated into its conjugate base. The species present are represented in the equation: $H_2X + NaOH \rightleftharpoons NaHX + H_2O$.
 - d) At midway to the second equivalence point, half of all of the protons in the solution (from both reactions) have reacted. The species present are represented in the equation: $NaHX + NaOH \rightleftharpoons Na_2X + H_2O$.
 - e) At the second equivalence point, both reactions will have gone to completion. This means that all of the protons in both reactions have dissociated. The species present are represented by the equation: $H_2X + 2NaOH \rightleftharpoons Na_2X + 2H_2O$

Discussion:

The primary source of error found in this experiment is the determination of the endpoint. Once the endpoint of the experiment was reached, the solution very quickly progressed past the endpoint if the burette was not closed immediately. The solution very frequently turned a darker pink after a few seconds, which indicated that the experiment had gone past the endpoint, and therefore the number of moles of the acid and base were not equal to each other. The opening and closing of the burette had to be done manually, and as this was difficult to do while trying not to disturb the other parts of the apparatus or affecting the drip count in any way.

Another point at which error may have occurred is the calibration of the drip counter, and the recording of volume thereafter. While the drip counter was calibrated according to the instructions, the volume readings of the drip counter were substantially different than the readings of the volume that were obtained visually. The drip counter was nudged several times as the burette was opened and closed, and at several points even stopped counting drips. It is likely that the drip counter was not fully calibrated, which would account for the discrepancy in volume. The drip count was also changed completely towards the experiment by adjusting the top tap, because the top tap had been nudged so much that the drip count had changed from 1-2 drops per second to almost 6 drops per second. During the calculations, the amounts of volume that had been added from the burette was used instead of the volume recorded by the LabQuest because of the potential calibration issue. Since the visual value was used for the calculations, the calculations will state a different concentration of the standardized base solution than the graphs created with the LabQuest data, however the trends will be the same regardless of the method of recording because the rate was constant.

Human error may have also affected the accuracy of the experiment. The readings that were taken off of the burette are accurate to two decimal places. However, since the readings were relying on visual affirmation alone, the volume may have been off by several decimals, despite attempts to make it as accurate as possible. Without a mechanical counter for the volume of acid, there is no way for the experiment to be one-hundred percent accurate.

Environment at the time of the experiment is another factor of experimental error that needs to be considered. A change in pressure or temperature can slow down or speed up the

experiment, and the true equivalence point may be inaccurately demonstrated. As well, a change in environment can also affect the base in a negative way because if the air is more humid, and has a higher water content, the base will react more readily with the air. This will cause more water to be absorbed, the concentration of the base to decrease, and the value for the concentration of the base will become inaccurate.

Conclusion:

The values obtained for the concentration of the dilute solution and the concentration of the unknown acid were 0.12M and 0.06M respectively. These values are slightly inaccurate, because, as the graphs demonstrated, the true value of the equivalence point was slightly higher than the recorded value. The average pH at the equivalence point for the solution during the standardization of the base was 11.19, and the average value for the pH at the equivalence point for the solution with the unknown acid was 11.34.

References:

Eddy, D. (n.d.). Retrieved from <http://www.chem.latech.edu/~deddy/chem104/104Standard.htm>

Acid-Base Titration reaction. (n.d.). Retrieved from [http://www.westfield.ma.edu/cmasi/gen_chem1/Solutions/reactions in solution/solution stoichiometry/titration.htm](http://www.westfield.ma.edu/cmasi/gen_chem1/Solutions/reactions%20in%20solution/solution%20stoichiometry/titration.htm)

Titration Curves. (n.d.). Retrieved from http://www.chemistry.uoguelph.ca/educmat/chm19104/chemtoons/titration_curves.htm

Raw Data:

Lab 4 OCT 31 2018

STARTING THE EXPERIMENT

- ↳ 250mL from bottom of meniscus of distilled water
- ↳ 5mL (from bottom of meniscus) of 6M of NaOH
- ↳ when NaOH poured in initially coated oily texture/appearance
- ↳ no effect after 2 mins

CALIBRATING THE DROP COUNT

- ↳ 2.6mL added to graduated cylinder
- ↳ final volume in graduated cylinder was 4.8mL after the addition of 36 drops

STANDARDIZING THE DILUTED NaOH SOLUTION

~~MONOPROTIC~~
~~UNIONIZED~~ ~~2.6~~ ~~36~~ ⇒ Concentration = 0.100M

TRIAL 1

- ↳ No initial buret volume is at ~~2~~ the ^{2.00mL} 2.00mL, so there is 23mL in the buret
- ↳ The exact final reading on the buret is 12.00mL as the exact volume transferred is 10.00mL
- ↳ added distilled water until solution was at 100mL marked line
- ↳ when colour change of acid solution occurred; ^{volume of} base added ⇒
- ↳ ~~throughout~~ volume measured = 23.00mL, volume observed = 10.00mL
- ↳ final pH was 11.16 after the addition of ^(23.00mL) 23.00mL and ^(10.00mL) 10.00mL

TRIAL 2

- ↳ exact final reading of buret was 22.10mL (from an initial of 12.00mL), exact volume of acid transferred was 10.10mL
- ↳ added distilled water was until solution was at 100mL
- ↳ when ~~color~~ colour change of solution occurred, lab buret reading was 24.00mL, visual reading was ~~30~~ 10.00mL added
- ↳ final pH was 11.44 after the addition of ~~30.31~~ 24.00mL (lab buret) and 10.00mL (visual)

2/1

TRIAL 3

- ↳ initial reading of buret was 11.00 mL
- ↳ final reading was 21.00 mL, so the exact volume transferred was 10.00 mL
- ↳ added distilled water until solution was at 100 mL
- ↳ when colour change of solution occurred, LabQuest reading was 25.60 mL, visual reading was 15.00 mL
- ↳ final pH was 11.54 after the addition of 35.73 mL (Lab Quest) and ~~12.00~~ 12.00 mL (visual)
- ↳ different because drop rate changed

TRIAL 4

- ↳ initial reading of buret was 11.00 mL, final was 21.00 mL
- ↳ exact volume transferred was 10.00 mL
- ↳ added distilled water to 100 mL line
- ↳ colour change occurred after 25.20 mL (Lab Q) or 9.00 mL ^{was added} (visual)
- ↳ final pH was 11.33 after addition of 32.13 mL (Lab Q) or 12.00 mL (visual)

DETERMINING CONCENTRATION OF UNKNOWN ACID \rightarrow ACID #2

TRIAL 2

- ↳ initial reading of buret was 2.00 mL final was 12.00 mL
- ↳ exact amount transferred was 10.00 mL
- ↳ added distilled water to ~~100.00~~ 100 mL mark
- ↳ colour change occurred after 25.87 mL (Lab Quest) and 10.00 mL (visual)
- ↳ pH final was 11.46 after the addition of (Lab Quest) and 34.00 mL (visual) 12.00 mL

TRIAL 2

- ↳ initial buret reading was 12.00, final 22.00, transferred 10.00 mL
- ↳ added up to 100 mL line distilled H₂O

OK

↳ colour change occurred after the addition of 25.73 mL (Lab Quest) and 10.00 mL (visual)

↳ final pH was 11.66 after the addition of ^{39.60} 45.73 mL (Lab Quest), 16.00 mL (visual)

TRIAL 3

↳ initial buret reading was 11.00 mL, final was 21.00 mL

↳ total exact volume added was 10.00 mL

↳ added distilled H₂O to 100 mL line

↳ colour change occurred after 27.20 mL (Lab Quest) or 10.00 mL (visual) was added

↳ final pH was 11.66 after the addition of 42.53 mL (Lab Quest) or 14.00 mL (visual)

TRIAL 4

↳ initial buret reading was 10.00 mL, final was 20.00 mL

↳ exact volume added was 10.00 mL

↳ added distilled water to 100 mL line

↳ colour change occurred after 26.67 mL (Lab Quest) or 10.00 mL (visual) was added

↳ final pH was 11.34 after an addition of 32.55 mL (Lab Quest) or 16.00 mL (visual)

BAH