***Experiment 2 Lab Report: Volumetric Analysis***

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**Date: 10/31/16**

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**Please use this template. If you use this template and write in the indicated sections, the lab report should be a maximum of 8 pages (or 10 pages if you need to use the optional pages); you may delete instructions in red font. Font should be 12 point, Times New Roman for text; font can be 8 points for equations. Use single-spacing. Do not adjust margins. Save this document with filename “Expt2\_Author\_TAname”, where Author is your name, and TAname is your TA’s name.**

**ACCURACY (20 pts)**

Unknown #: 69

Molecular Mass (g/mol), including standard deviation: 152.9 ± 0.4 (g/mol)

*p*Ka, including standard deviation: 3.6 ± 0.4

ID of unknown: Mandelic Acid

**ABSTRACT (20 pts)**

***Write your own abstract here (should be no more than 130 words). Remember that your result, including error, must be stated in the abstract.***

In this experiment, volumetric analysis was used to determine the species of an unknown weak organic acid. Based on the results and the calculation, the predicted species of weak monoprotic acid is Mandelic Acid. The calculated molar mass of the unknown was 152.9 ± 0.4 (g/mol) while the actual molar mass was 152.15 (g/mol). The Calculated pKa was 3.6 ± 0.4, while the actual value was 3.37. In this experiment, KHP was used to standardize a solution of NaOH, which was then used to titrate a known mass of an unknown species of acid to calculate the molar mass. The titration was repeated with a pH meter to find the pKa of the unknown acid. The two values were then used to determine the species.

**INTRODUCTIONis NOT required for this lab report (0 pts)**

**EXPERIMENTAL is NOT required for this lab report (0 pts)**

**RESULTS (50 pts total = Data + Observation + Calculations)**

***Write your results below in the appropriate spaces.***

IMPORTANT: The experimental data and observations reported here, in the “Results” section, must also be recorded in your laboratory notebook to be valid. It is unacceptable to rely on memory. Do not attempt to report data/observations that were never recorded in your notebook, and do not add content to your laboratory notebook after the day of the experiment. These acts of falsification and altering of the notebook are violations of academic and scholarly integrity. If you forgot to record information on the day of the experiment, you can indicate: “not recorded in notebook” in the relevant space below.

**RESULTS: Data (12 pts)**

Recorded mass of KHP and volume of NaOH to reach end point of KHP solution (Part II):

|  |  |  |  |
| --- | --- | --- | --- |
|  | **Trial #1** | **Trial #2** | **Trial #3** |
| Mass of KHP (mg) | 609.6 | 602.1 | 604.2 |
| Volume of NaOH (mL) | 34.22 | 34.73 | 34.40 |

Recorded mass of unknown acid and volume of NaOH to reach end point of acid solution (Part III):

|  |  |  |  |
| --- | --- | --- | --- |
|  | **Trial #1** | **Trial #2** | **Trial #3** |
| Mass of unknown acid (mg) | 304.4 | 302.0 | 290.3 |
| Volume of NaOH (mL) | 23.10 | 22.95 | 22.12 |

Mass of unknown acid (mg) for complete titration curve (Part V, procedures 2-3): 1529 mg

Volume of **water** added to unknown acid (mL) for complete titration curve: 100 mL

Recorded pH as a function of volume of NaOH added to unknown acid for complete titration curve, Part V, procedures 2-3 (if additional rows are needed, you may complete the table on pg 9). You will insert the titration curve on page 6:

|  |  |
| --- | --- |
| **volume (mL)** | **pH** |
| **4.58** | 2.48 |
| **10.61** | 2.79 |
| **16.09** | 3.09 |
| **22.01** | 3.42 |
| **27.26** | 3.78 |
| **31.58** | 4.37 |
| **33.43** | 5.69 |
| **33.51** | 6.59 |
| **33.62** | 8.24 |
| **33.67** | 8.89 |
| **33.71** | 9.10 |
| **33.73** | 9.35 |
| **33.81** | 9.60 |
| **33.86** | 9.81 |
| **33.89** | 9.97 |
| **33.93** | 10.17 |
| **34.08** | 10.33 |
| **34.35** | 10.62 |
| **34.92** | 10.94 |
| **36.37** | 11.27 |
| **38.73** | 11.54 |
| **46.62** | 49.26 |
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**RESULTS: Observations (12 pts)**

***Describe your observations in the blank spaces below.***

IMPORTANT: Provide detailed observations for each component of the experiment as documented in your notebook (e.g., color of sample, volume and/or number of drops of chemicals added, unusual incidents, etc.). Do not rely on your memory. If you forgot to record information, you can indicate “not recorded in notebook” in the appropriate spaces below.

*NaOH standardization with KHP (Part II):*

* Used buret #30
* Small amounts of KHP crystals fell out of tray for trial 3

*Titration of unknown acid to determine molar mass (Part III):*

* Over shot a little on all three titrations

*Titration of unknown acid to determine pKa (Part V):*

* The first “quick and dirty” titration was missed, skipped directly to the measurement titration
* Messed up first titration (the titration was ended too early (as soon as the solution turned pink)
* The second titration had certain points where the pH jumped nearly 1 pH when one half-drop was added
* Third titration: the halfway point to the end was missed, resulting in no value for it
* Small amounts of NaOH solution leaked into the unknown solution at the end of the titration
* The acid solution was over heated and not cooled before addition into volumetric flask, the water contracted and more DI water was added to fill to the calibration line.

**RESULTS: Calculations (26 pts)**

***The beginning part of this section is started for you. Show your calculations in the blank spaces below. You must utilize an equation editor for this section.***

Standardization of NaOH: The determination of the concentration of NaOH was based on the knowledge that moles of NaOH = moles of KHP. The general formula is given:

Determination of molar mass of unknown: The determination of the molar mass of the unknown acid was based on the knowledge that moles of organic acid = moles of NaOH. The general formula is:

Determination of *p*Ka of unknown acid: The Henderson Hasselbalch equation is used to determine the *p*Ka of the unknown acid:

*Standardization of NaOH (show a calculation for one trial)*:

Calculation for trial #1

*Determination of molar mass of unknown acid (show a calculation of one trial):*

Calculation for trial #1

*Determination of pKa of unknown acid (show calculations for towards end point):*

1/3:

1/2:

This titration point was missed, there is no possible calculation for this point in the titration

2/3:

Calculation of the average molar mass and pKa, and the corresponding standard deviations:

Molar Mass trials:

1: 153.2 (g/mol)

2: 152.9 (g/mol)

3: 152.5 (g/mol)

Titration trials

pKa1 = 3.38

pKa2 = 3.35

pKastart = 3.99

pH = -log([H+])

[H+] = 0.003

pKastart = 3.99 = 2.48 – log

3.57 =

Average molar mass = 152.9 g/mol ± σ = 0.4 g/mol

Average pKa = 3.57 ± σ = 0.4

**DISCUSSION (30 pts)**

This experiment was carried out to determine the identity of an unknown species of organic acid. The experiment utilized a buret for accurate and controlled additions of standardized NaOH solution to determine the molar mass and the pKa of the unknown species. The particular sample number used in this experiment was sample # 69 and the calculated molar mass of the species is 152.9 ± 0.4 (g/mol) and the calculated pKa is 3.6 ± 0.4. Based on these results, the species is most likely Mandelic Acid.

This experiment had many areas where error emerged, especially in the portion with the titration to determine the pKa. The first portion, where the NaOH was standardized, small portions of the KHP fell out of the weighing boat as it was being added to the third flask for trial #3. This drove the average molarity of the solution standardization calculation down. And this may have resulted in a molarity that was lower than the actual molarity. The standard solution had a concentration of 0.0860 ± 0.0009 M the percent standard deviation was 0.11%, indicating at least that the molarity was precise. Next, in the titrations determining the molar mass of the unknown species, in all three titrations, there was overshooting. The over shooting in the three titrations led to the species having a higher calculated molar mass than it should have. Hence, the calculated molar mass was 152.9 (g/mol) when the actual molar mass was 152.15 (g/mol). The third analysis step, where the pKa of the unknown species was determined gave the most trouble. In the titration of the weak acid, the “quick and dirty” first titration was forgotten, and the first titration was done in the 0.3 increments. That titration was thrown out at the equivalence point and not the end point, leading the results of that experiment to be useless. In the second try on the titration with the recording of individual pH values with 0.3 increments, there was one value, where the pH jumped up by 1.3 after the addition of one drop. This may have led to an inaccurate estimation of the endpoint, where the slope at that point had a massive spike. In the third titration, the halfway point was missed, meaning that the pKa could not be calculated at that point. In its place, a pKa estimation was used based on the starting pH (detailed in the previous calculation section). There was no left over 0.1 M acid left over after the third titration because 25 mL of it was invested into cleaning and prepping the 25mL volumetric pipette. There was no time left to make another 100 mL of the 0.1 M weak acid.

In this lab, the majority of the mistakes were made in the third portion of the lab, resulting in a relatively large percent standard deviation %RSD = 11% = (0.4/3.6)x100 . This inaccuracy was caused by the need to use to use a replacement value for the halfway point, which would have been where the pKa would have equaled the pH, this was caused by disorganization in the assignments of stop points for the Base solution. As for improvements to the overall procedure. If funding permits, increase the size and proportions of the reagents, this will result in smaller errors and some errors, like overshooting to be minimized. This is especially true with the final portion, where there should really be more than 100 mL of the 0.1 Acid solution prepared, especially if there were 3 trials recommended in the procedure. If students were to have trouble and commit enough errors to make more than one trial unusable, there would be enough acid left for more trials.