Identifying an unknown acid

Objective:

The objective of this experiment is to prepare and standardize a sodium hydroxide solution and use it to determine the **equivalent weight, pKa,** and **melting point** of an unknown acid. Using this information the identify of an unknown acid will be determined.

## **Introduction**

## **Titration:** Titration refers to the addition of a solution of one reagent, usually from a burette, to a known amount of the other reagent, and then determining the volume of solution added when the reaction is complete.

**grams of acid/moles of H+ reacted.** The number of moles of *H+* is equal to the number of moles of OH- from the base.

**equivalent weight of an acid:** I**t** is defined as the weight of acid that provides one mole of H+ in the reaction. For a monoprotic acid like HCl, the equivalent weight and the molecular weight are the same.

## **Acid Strength, pKa:** As a measure of the relative strength of an acid, chemists use a measurement known as the **acid dissociation constant,** which usually has the symbol *Ka.* The acid strength of acids, HA, is a property that can be used to explore the relationship between structure and chemical properties.

**What is a primary standard?** A primary standard is a soluble solid compound that is very pure, with a consistent formula that does not change on exposure to the atmosphere, and has a relatively high molar mass.

**What was the primary standard in this experiment?** In this experiment, the primary standard used is the oxalic acid

**If you didn't use a primary standard, indicate what it should have been?** If primary standard is not used, we might get an error in the concentration of the secondary standard which may give us wrong results.

**Why were there 2 standard solutions?** In order for a titration to be accurate, the base solution must first be standardized

**That is, why wasn’t NaOH(aq) used as a primary standard for titrating the unknown acid?** Sodium hydroxide solutions cannot be prepared with adequate precision simply by weighing out solid NaOH, since the commercial material is not sufficiently pure and absorbs water and CO2 quickly from the air.

**How would melting of a pure sample differ from melting of an impure sample?**

A *narrow range* of one to two degrees Celsius implies a high degree of purity, while a *broad range* usually implies an impure.

**What is buffer?** buffer is a mixture of a weak acid and a conjugate base in roughly equal molar proportions. One property of a buffer is that its pH is changed only slightly by adding strong acid or strong base to it. For this reason, as you titrate your unknown acid, the pH will only change very slowly.

**For a weak acid, under what conditions will pH = pKa?**

When the titration of the weak acid with base reach half the equivalence volume, the pH at this point is equal to the value of pKa.

**Procedure:**

## **Preparation of the Standard Oxalic Acid Solution**

1. Weigh a sample of the hydrate that is between 0.3- 0.5grams Tare a clean, dry beaker on the analytical balance , add an appropriate amount of oxalic acid, and weigh the beaker containing the acid
2. Transfer all of the acid to a 250.0 mL volumetric flask, fill the volumetric flask half-full with water and swirl contents until the acid is all dissolved. When you are certain that all of the solid has dissolved, add water to the mark, stopper the flask and invert several times to mix thoroughly.
3. Calculate the concentration of the oxalic acid following the equation below:

[oxalic acid]=moxalic acid/Moxalic acid x V

Register the data in the lab worksheet

## **Standardization of NaOH**

1. Prepare one liter of approximately 0.10 *M* NaOH by dilution of the stock solution provided. Mix it thoroughly and store in a properly labeled plastic bottle. Be sure to record the original concentration of the stock solution, the amount of water that you used to dilute the stock solution and the final concentration of your NaOH solution.
2. Using a volumetric pipet, transfer 25.00 mL of the prepared oxalic acid solution into a 125 mL Erlenmeyer flask. Add two drops of phenolphthalein indicator to the acid solution. Fill the burette with your NaOH solution and titrate the acid in the flask with the NaOH from the burette. The titration is complete when the first pink color persists in the flask. Swirl the flask during the addition of base to ensure complete mixing**.**
3. Repeat the titration 2 more times so that you will make 3 trials**.** Calculate the concentration of the base for each trial, and obtain a mean and standard deviation. The average concentration that you calculate is the concentration of your now standardized NaOH.

The reaction that occurs bet the sodium hydroxide and the oxalic acid is written as followed:

H2C2O4(aq) + 2 **NaOH**(aq) → Na2C2O4(aq) + 2 H2O(l)

## **Molar Mass of a Monoprotic Unknown Acid**

1. On the analytical balance, measure out approximately 0.30 g of your unknown **acid.**
2. transfer to a 250 mL Erlenmeyer flask which has been cleaned but not necessarily dried. Add 50-100 mL of water as well as two drops of phenolphthalein indicator to the flask.
3. If the acid does not dissolve easily, you may need to heat the mixture gently. If most, but not all the acid dissolves , you may proceed with the titration, as the conjugate base of the acid created during the titration will be quite soluble in water.
4. Clean, rinse and condition a 50-mL buret with standardized approximately 0.10 *M* sodium hydroxide solution. Be sure to record the concentration of the NaOH in your lab work sheet. Fill the buret to somewhere near the top and titrate your sample to phenolphthalein end­ point.
5. Repeat the procedure three times. To be sure your results are good, calculate the ration of grams of unknown used to volume of titrant used. The ratios should agree within 1%. At this point, you may want to calculate the GMM of your unknown acid from the three titrations and calculate the average of your values.

## **The Melting Point**

1. Place just a few small crystals of your unknown acid in the center of the top of your melting point apparatus.
2. Allow the temperature to increase fairly slowly and record the temperature at which it melts.
3. Once you have an approximate melting point, now you will measure the melting point much more carefully by approaching the melting point very gradually.
4. Repeat the the experiment so that the total will be 3 trials then record the data in the work sheet.

**Measuring pKa**

Measure out another sample of the unknown acid on the analytical balance.

Using the Vernier Interface

1. Calibrate the pH electrode using two buffer solutions of known pH.
2. Using the calculated GMM of the unknown acid obtained from your earlier titration, calculate the volume needs to titrate it.
3. Divide the volume by two and add this volume to your sample.
4. Measure the pH using the calibrated pH meter. This will be the pKa of your unknown.

Repeat the process to get an average pK**a**

3

**Sample Data sheet**

mass of oxalic acid : 0.84g

volumetric flask**:** 250 mL

Standardization of NaOH solution

**Standard solution preparation**

Initial concentration of stock solution of NaOH: 1M

Pipet size: 25 mL

Amount of water used for solution: 225 mL

Approximate concentration of the standard solution: 0.1M

**Standardization of NaOH solution**

|  |  |  |  |
| --- | --- | --- | --- |
|  | **Trial 1** | **Trial 2** | **Trial 3** |
| Initial buret reading (mL) | **0.2** | **1.15** | **0.55** |
| Final buret reading (mL) | **28.4** | **27.85** | **27.29** |
| Volume of base used (mL) | **28.2** | **26.7** | **26.74** |

**Melting point of unknown acid**

|  |  |  |  |
| --- | --- | --- | --- |
|  | Trial 1 | Trial 2 | Trial 3 |
| Melting point range (°C) | 124 | 123.2 | 123.3 |
| Average melting point (°C) | 122.3 | | |
| Proposed literature melting point (°C) | 122.3 | | |

**Titration of Unknown Solid Acid**

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
|  | **Trial #1** | **Trial #2** | **Trial #3** | **Trial #4** |
| Initial buret reading (mL) | 0.4 | 0.1 | 0.35 | 0.25 |
| Final buret reading (mL) | 15.85 | 16.6 | 16.55 | 17.55 |
| Volume of base used (mL) | 15.45 | 16.5 | 16.2 | 17.3 |
| Mass of unknown (gram) | 0.187 | 0.2 | 0.1975 | 0.21 |

Sample calculation Sheet

**Standard Oxalic Acid Solution**

|  |  |
| --- | --- |
| Mass of oxalic acid | 0.84 g |
| Moles of oxalic acid | 0.0066 moles |
| Volumetric flask | 250 mL |
| Molarity of oxalic acid | 0.0266 |
| Molarity of H+ ions in oxalic acid | 0.0533 |

**Standardization of NaOH Solution**

|  |  |  |  |
| --- | --- | --- | --- |
|  | **Trial #1** | **Trial #2** | **Trial #3** |
| Moles of oxalic acid (moles) | 0.00133 | 0.00133 | 0.00133 |
| Volume ofNaOH (mL) | 28.2 | 26.7 | 26.74 |
| Molarity ofNaOH | 0.095 | 0.0996 | 0.0994 |
| Average molarity (M) | 0.098 | | |

**Determination of equivalent weight**

|  |  |  |  |
| --- | --- | --- | --- |
|  | **Trial #1** | **Trial #2** | **Trial #3** |
| Mass of unknown (gram) | 0.183 | 0.1957 | 0.1934 |
| Volume of NaOH (mL) | 15.45 | 16.5 | 16.2 |
| Moles of NaOH (moles) | 0.0015 | 0.0016 | 0.00158 |
| Moles H+ reacted (moles) | 0.0015 | 0.0016 | 0.00158 |
| Equivalent weight (gram) | 120.86 | 120.59 | 122.008 |
| Average equivalent weight | 121.15 g/mol | | |
| Standard deviation of molar mass | 0.75 g/mol | | |
| Proposed literature value of molar mass | 122 g/mol | | |
| % error of molar mass | 0.69% | | |

**Determination of pKa**

|  |  |  |  |
| --- | --- | --- | --- |
|  | **Trial 1** | **Trial 2** | **Trial 3** |
| **Mass of Unknown (g)** | **0.21** | **0.23** | **0.19** |
| **Calculated volume of NaOH at equivalence** | **17.55** | **19.2** | **15.87** |
| **Calculated of the volume of NaOH at half equialence** | **8.77** | **9.6** | **7.93** |
| **pH of the solution at half equivalence** | **4.17** | **4.18** | **4.2** |
| **Average pKa** | **4.183** | | |
| **Standard deviation of pKa** | **0.015** | | |
| **Proposed value of pKa** | **4.2** | | |
| **% error of pKa** | **0.39%** | | |

**Lab report summary**

|  |  |
| --- | --- |
| **Standard concentration of NaOH** |  |
| **Average molar mass of unknown** | **121.15** |
| **Average melting point of unkown** | **123.5** |
| **Average pKa of the unknown** | **4.183** |
| **Unknown acid identity** | **Benzoic acid** |