

## Investigation 5: Titration of a Weak Acid

**Focus Questions:** *How can we use titration data to determine the identity of an unknown monoprotic acid?*

### Pre-lab required reading

Chemistry; an Atoms-Focused Approach: Sections [15.1 – 15.6](#), [16.4](#)

Primers:

[Volumetric glassware use - Buret](#)

[Volumetric glassware use – General](#)

[Volumetric glassware use – volumetric flask](#)

[Volumetric glassware use – volumetric pipet](#)

### Safety and Waste Disposal

- Eye protection should be worn at all times.
- All solutions used in this investigation can be washed down the drain with copious amounts of water.

### Background

Molar mass and  $pK_a$  are distinguishing characteristics of weak acids. The experimental determination of molar mass and  $pK_a$  provides a way to identify an unknown weak acid.

Titrating the acid with a solution of a strong base (such as NaOH) of known concentration provides a practical way to determine the acid's molar mass and  $pK_a$ . In order to accomplish such a determination, the base must first be **standardized**, that is, that the concentration of the base to be used must be experimentally ascertained. Standardization is performed by using the strong base to titrate a known mass of a **primary standard** – a stable compound that is available in very high purity.

In this experiment, you will first make a solution of NaOH and then standardize it by using it to titrate a primary standard, potassium hydrogen phthalate ( $KHC_8H_4O_4$ , familiarly known as “KHP”). Then, you will use the NaOH to titrate a known mass of an unknown acid; this will allow you to compute the acid's molar mass. Finally, you will prepare a mixture that contains equal concentrations of the weak acid and its conjugate weak base; this will allow you to determine the  $pK_a$  of the acid. Comparison of the experimental results to the known molar masses and  $pK_a$  values of possible weak acids will allow you to identify your unknown acid.

### Terminology

standardize: to experimentally determine the concentration of a solution to a relatively high accuracy

primary standard: a stable compound of very high purity used to determine the concentration of a laboratory solution

### Procedure

#### Make a sodium hydroxide solution

Calculate the amount of concentrated NaOH (50.5% by weight,  $D = 1.53\text{g/mL}$ ) needed to make 500mL of 0.1M NaOH. Measure this amount into a 500-mL volumetric flask and dilute to the mark. Add your solution to the carboy for your section to use for the following of experiments.

### Standardization of a sodium hydroxide solution

Place 0.4 - 0.6 g (be sure to record the precise mass) of dried potassium hydrogen phthalate (stored in the desiccator) in a clean 250 mL Erlenmeyer flask and dissolve the salt in about 75 mL of DI water. Fill a cleaned and conditioned buret with sodium hydroxide solution that was prepared by your lab section (Note that all students must complete the preparation and add their solution to the carboy before you can begin this procedure.) Titrate the potassium hydrogen phthalate sample with the  $\text{NaOH}_{(\text{aq})}$ , using 3 – 4 drops of phenolphthalein as indicator, to a pale pink endpoint. Repeat the titration until you have three titrations that yield a concentration of sodium hydroxide that are within 5% of each other.

### Determination of molar mass

Place about 0.3 – 0.4 g (be sure to record the precise mass) of your unknown acid in a clean 250 mL Erlenmeyer flask and dissolve the salt in about 25 - 50 mL of DI water. Fill a cleaned and conditioned buret with sodium hydroxide solution. Titrate the weak acid sample with the  $\text{NaOH}_{(\text{aq})}$ , using 3 – 4 drops of phenolphthalein as indicator, to a pale pink endpoint. Repeat the titration until you have three titrations that yield a molar mass of unknown acid that are within 5% of each other.

### Determination of the pKa of an unknown weak acid

Perform one or both of the following procedures as directed by your instructor.

**Titration Method:** Place about 0.4 g (be sure to record the precise mass) of unknown acid in a clean 400 mL beaker and dissolve the acid in 75.0 mL of DI water. Fill a cleaned and conditioned buret with sodium hydroxide solution. Calibrate a pH meter using pH 4 and pH 7 standard buffer solutions. Place the electrode in the unknown acid solution. Slowly add the  $\text{NaOH}$  solution from the buret. After each addition of  $\text{NaOH}$ , let the pH reading stabilize and record its value. No region of the curve should have a jump in pH of more than 1 unit. You will need to be very careful (add just one drop of  $\text{NaOH}$  at a time) as you approach the equivalence point since the pH will increase rapidly at this point. (Note that you can use the results of the determination of the molar mass above to estimate where the equivalence point should occur). Stop titrating when the pH has leveled off (near 11) for at least 5 mL.

**Mixture Method:** Place about 1 g (be sure to record the precise mass) of unknown acid in a clean, dry 250-mL Erlenmeyer flask. Dissolve the unknown acid in 100.00 mL of DI water that has been measured using a 50.00-mL volumetric pipet. Using the volumetric pipet, transfer 50.00 mL of the weak acid solution into a second clean, dry 250-mL Erlenmeyer flask. Fill a cleaned and conditioned buret with sodium hydroxide solution. Titrate the weak acid in the second flask with the  $\text{NaOH}_{(\text{aq})}$ , using 3 – 4 drops of phenolphthalein as indicator, to a pale pink endpoint. To the first flask, add an amount of distilled water equal to the amount of  $\text{NaOH}_{(\text{aq})}$  added to the second flask. Mix the solutions in the two flasks together and measure the pH of the resulting solution.

## References

Atkins, P.; Jones, L. "Chemical Principles: The Quest for Insight", 6th ed.; Freeman: New York. **2013**.