Experiment 1
Acid-Base Titrations

Discussion

Volumetric procedures are among the most common and convenient methods of analysis. The preparation of a reactive solution of accurately known concentration is fundamental to these methods, and the exercise serves as an introduction to the techniques of solution preparation and titration.

The objective of this exercise is to prepare and accurately determine the concentration of a solution of NaOH, and to use that standardized solution in the determination of the concentration of acid in a commercially available sample. The first step is the preparation of a sodium hydroxide solution whose concentration is approximately known. The second step is the determination of the concentration accurately by titration with a solution containing a known concentration of a **primary standard**, in this case potassium hydrogen phthalate. (Primary standards are substances which may be obtained in a stable form of known purity and which react with other substances quickly in a definite and known manner.)

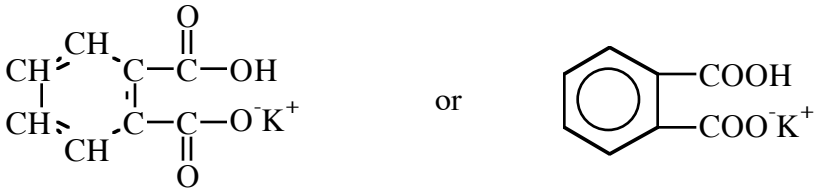
Potassium hydrogen phthalate and sodium hydroxide react as follows:

$$KHC_8H_4O_4 + NaOH \rightarrow KNaC_8H_4O_4 + H_2O$$

or, expressed as an ionic equation:

$$HC_8H_4O_4^- + OH^- \rightarrow C_8H_4O_4^{2-} + H_2O$$

The complete structure of potassium hydrogen phthalate is



This reaction is a representative of an acid-base reaction. In this case the hydrogen phthalate ion is the acid (proton donor) and the hydroxide ion is the base (proton acceptor). The stoichiometric ratio between the hydrogen phthalate ion and the hydroxide ion, and therefore between the potassium hydrogen phthalate and the sodium hydroxide, is obviously one to one.

The equivalent weight of an acid (also called an **equivalent**) is the weight in grams that furnishes one mole of H^+ ion in an acid-base reaction. Correspondingly, **the equivalent weight of a base** is the weight in grams that accepts one mole of H^+ ion in an acid-base reaction. (In the reaction under consideration, what is the equivalent weight of the potassium hydrogen phthalate and the sodium hydroxide?)

In determining the exact concentration of a solution, a procedure called a titration is commonly used. A **titration** is a process in which a solution containing a known amount of a substance is allowed to react with a second solution containing an unknown concentration of another substance that will react with the first substance in a known and reproducible manner. The substances are allowed to react until there is some indication that equivalent amounts of the substances have reacted. The solutions are measured from a **buret**, a long, graduated glass tube with a stopcock at the bottom.

In the present case a solution of potassium hydrogen phthalate is prepared in an erlenmeyer flask by dissolving an exactly known weight of pure potassium hydrogen phthalate in water. The sodium hydroxide solution (whose exact concentration is unknown), is delivered from a buret until an amount equivalent to the amount of potassium hydrogen phthalate has been added. This point in the process is called the **equivalence point of a titration**.

We can monitor the progress of acid-base titrations by two means. The first uses a pH meter, and the second uses an acid-base indicator. An indicator is a dye that has the particular property of changing color as a function of pH. You will select an appropriate indicator to use in your titrations based on the data you obtain using a pH meter. The point in the titration when the indicator changes color is called the **end point**. Ideally the indicator should be selected so that the end point coincides with the equivalence point.

Procedure

(For instructions on using the pH meters, see below)

Using the ~9M NaOH solution provided, make up approximately 500 mL of ~0.1M NaOH in a polyethylene bottle. Cap tightly and shake thoroughly.

You will standardize your NaOH solution against potassium hydrogen phthalate. Accurately weigh by difference 4 separate 0.20-0.30 g portions of dry potassium hydrogen phthalate into clean and dry 150 mL or 250 mL beakers. Add approximately 50-100 mL distilled water to each and swirl until the potassium hydrogen phthalate is completely dissolved.

Do a rough titration (Titration 1), adding approximately 1 mL of your NaOH solution at a time, recording the pH after each addition (be sure to give the reagents a few seconds to fully react before recording the pH.)

In recording data from a titration, record the actual buret readings (estimate to nearest 0.01 mL), not the volume of titrant delivered. This eliminates a possible source of error.

Make a rough plot of your titration data. Based on your titration curve, select an appropriate indicator and add a few drops to one of the three remaining flasks. Titrate this sample more carefully (Titration 2) – recording both the pH meter as well as the indicator. You may add approximately 0.5 mL at a time in the "flat" regions of the titration curve, then add 1 drop at a time as the pH begins to change more rapidly close to the equivalence point. You should determine the equivalence point to within a drop of titrant.

Record the color change along with the volume of NaOH used and the pH and check that the chosen indicator accurately marks the equivalence point of your titration.

If that is the case, add your indicator to the remaining two samples and titrate those (Titration 3 and 4) without the help of the pH meter, only using the indicator. Again, determine the equivalence point to within a drop of titrant.

Check the precision of your results. Disregard the first (rough) titration and calculate the ratio of the volume titrant added to the weight of the sample for the three careful titrations. Determine the average of these ratio values, their standard deviation (absolute error), and the relative error. A relative error below 0.5% could be considered a satisfactory precision in this experiment.

Compute the molarity of your sodium hydroxide solution. This solution will now be used to titrate a commercially available acid. You should take a 1 mL portion of the vinegar provided at your bench, dilute with about 50 mL of distilled water and titrate your sample carefully with the pH meter. Make sure to determine the equivalence point to within a drop of titrant. Prepare a titration curve and calculate the molarity of the acid and the concentration in weight % (assume a density of 1 g/mL). Compare your result to the claim on the bottle label.

Disposal

Your NaOH solution should be stored, tighly capped in your locker, for use in experiment 4.

All titration solutions can be washed down the drain with plenty of water. Any excess dry potassium hydrogen phthalate can be returned to the containers at the back of the lab.

Operation of pH Meter

A pH meter is essentially a voltmeter of extremely high internal resistance (many megaohms). The electrode couple generally used for the measurement of pH consists of a calomel reference electrode (whose potential is constant and reproducible) and a glass electrode whose potential is a linear function of the pH of the solution. Thus the electromotive force of the cell comprised of the couple will also vary linearly with the pH. The meter must first be calibrated using a standard buffer solution.

A. When you enter the labs, the probe electrodes will be in a buffer solution since they need to equilibrate for a while before you use them. The meters will already have been standardized for you, but before you start, you might want to check the calibration with the pH=4.0 buffer. Please use a different beaker to collect electrode rinsings and do not discard the buffer unless it becomes contaminated.

B. Leave the "function" knob on **standby** except when actually measuring a solution pH.

C. Lift the electrode from the standard solution, rinse it into another beaker, and carefully place it in the test solution (described below), leaving at least 1 cm clearance between the stir bar and the probe. Be sure that the stir bar does not hit the electrode. Stir slowly, both to protect the electrode and to avoid generating bubbles. (Wild fluctuations in the reading usually suggest the presence of bubbles around the electrode.) Turn the function knob to "pH," allow the readings to stabilize (about 30 sec), and read the meter.

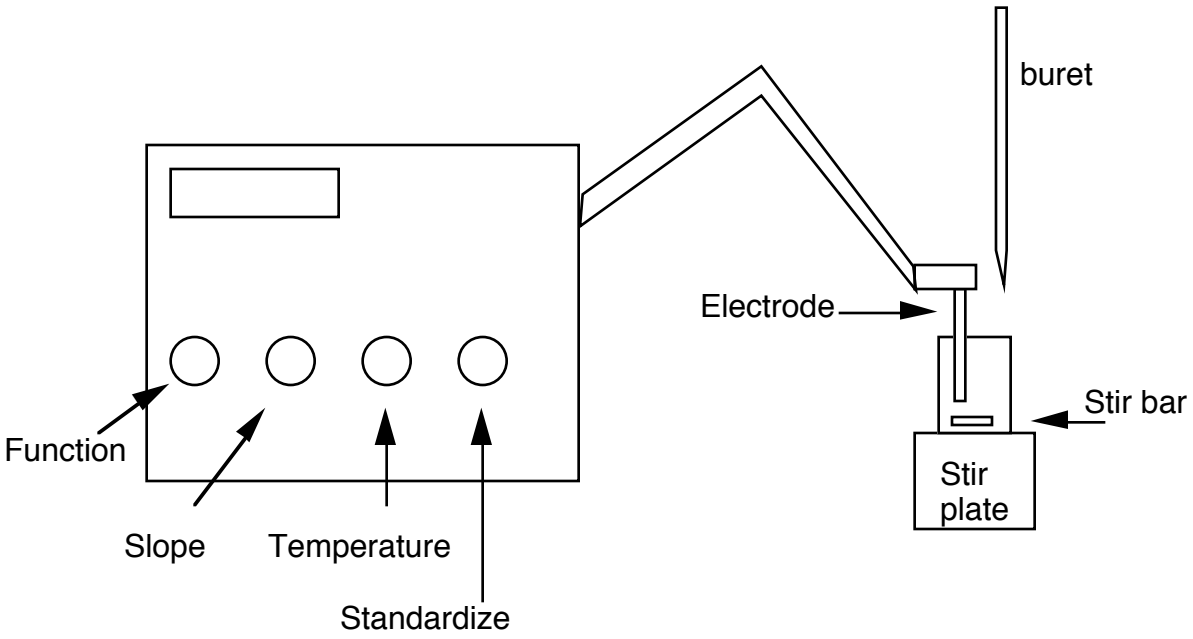


Figure 1. Set-up for titration using the Acumet pH meter.

Experiment 1 Worksheet — Acid-Base Titrations

Name_____ Date of Experiment: ____/____/____

Date of Report: ____/____/____

Standardization of NaOH solution against potassium hydrogen phthalate

Molarity of NaOH: _____ ± _____ (± _____ %)

(average) (std. dev.) (rel. error)

Prepare a titration curve (you can use a computer graphing program if you choose) for your careful titration following the guidelines in the introductory pages and turn it in with your worksheet. Mark the equivalence point and its coordinated on both axes.

Please attach your analysis: Report your initial data (weight of samples, volume of titrant used). Introduce symbols for your numerical quantities and use those in equations to indicate the mathematical transformations performed for every step in your analysis. Calculate and report the standard deviation (error) along with the average of the molarity. See the sample analysis in the introductory section.

Discuss if the indicator you used was appropriate for your sample. Are your results reasonable?

Determination of concentration of commercial acid

Commercial acid sample: _____

Identity of acid in sample: _____

Concentration of acid according to label: _____

Molarity of commercial acid: _____ ± _____

Concentration of acid in weight %: _____ ± _____

Attach a titration curve for the commercial acid and show your results and calculations on a separate sheet. Follow the guidelines given above.

For the discussion, you could consider the following questions. Does your result agree with the bottle label? Does your result seem to make sense? Is the manufacturer cheating? Any comments about possible discrepancies?