EXPERIMENT 7

Identifying a Substance by Acid-Base Titration

SAFETY WARNING

In this experiment you will be working with NaOH pellets and using 0.25 M NaOH as a titrant. Sodium hydroxide is extremely basic, caustic, and corrosive. Use of rubber gloves when preparing or titrating with it is recommended. *NEVER* pour a solution of NaOH or any other caustic substance above eye level. Place the burette stand on the floor or on one of the pull-out shelves to fill your burette. If you get any of the solution on your hands or clothes, *rinse it off immediately with copious amounts of water*. If you spill a large amount on your clothing, *head immediately for one of the showers*. *Keep your hands away from your eyes at all times* during the experiment unless you have just rinsed your hands thoroughly. Sodium hydroxide solutions feel slimy or slippery to the touch. If your fingers ever feel extra slippery or if some part of your body starts itching, don't stop to ask a TA about this, *wash the affected part with lots of water immediately*. Stay alert and work in a controlled and *very neat* manner. Keep things clean at all times.

USE FRESH *DEIONIZED* WATER THROUGHOUT THIS EXPERIMENT

Deionized water should have had most of the carbon dioxide removed from it during its preparation. Solid NaOH and its solutions absorb carbon dioxide (an acid anhydride) from the air to form carbonate. The equilibrium level of dissolved CO_2 in water open to the atmosphere is about 1.5×10^{-5} M. This reduces the titer of the base, and also makes endpoints slow and less distinct. Fine powder that doesn't dissolve quickly when you are preparing the NaOH titrant is probably sodium carbonate. If you see white particles while preparing the NaOH the solution, it should be filtered through a Buchner or fritted-glass funnel and then transferred into a 1-L plastic bottle prior to use. See the Teaching Assistant.

UNKNOWN

Submit a clean, labeled, and dry sample vial to the instructor so that your unknown acid can be issued. Your name, section number, and your locker number should be written legibly on this vial. Note that *the vial must be turned in at least 1 lab period before you plan to do the experiment* so that the Teaching Assistants will have time to prepare the unknown.

BACKGROUND

Acid-base titrations can provide valuable information about the nature and properties of an acid or a base. These titrations can be useful not only in determining the molecular mass and pK_a values, but also whether the substance is polyfunctional. *Polyfunctional* acids and bases have two or more acidic or basic functional groups; i.e., they may donate or accept more than one proton. The end point of an acid-base titration can be monitored by using colorimetric indicators or a pH meter and glass electrode.

INSTRUMENTATION

Corning Model 430 pH meter.
Combination pH and reference electrode.
Magnetic stirrer and stir bar.
Electrode holder.

PREPARATION OF SOLUTIONS

0.25 M NaOH

1. To prepare 1 L, carefully pipet approximately 18 mL (about 20 g) of ***highly caustic***, concentrated, carbonate-free, 50% NaOH solution into a 1-L plastic bottle containing 500 mL of fresh deionized water. The solution is somewhat viscous, so it is better to use a pipet with a large tip opening.

2. Screw the cap on the plastic bottle and swirl to mix thoroughly.

3. Add another 500 mL of deionized water and mix thoroughly.

4. Immediately rinse out the pipet thoroughly to remove all traces of NaOH, which could be hazardous to you or others and will etch the glass surface.

Keep the cap tightly screwed onto the plastic bottle except when transferring some to your burette in order to minimize absorption of atmospheric carbon dioxide. In similar manner, you should also have a burette cap on the top of the burette for the same reason.

0.2 M Potassium Acid Phthalate (KHP) Standard Solution.

Prepare 250 mL of 0.2 M KHP. This solution will be used to standardize the NaOH titrant that you prepared to be *approximately* 0.25 M.

1. Calculate the mass of KHP ($KHC_8H_8O_4$, 204.22 g/mol) needed to prepare exactly 250 mL of 0.2000 M KHP. Accurately (to 0.1 mg) weigh by difference a quantity of KHP (oven-dried at 110 °C for 1-2 hr) close to this amount into a small plastic weighing boat. It does not have to be the *exact* amount calculated, but should be reasonably near. **NOTE: *NEVER* transfer chemicals *inside* an analytical balance**.

2. Transfer the KHP quantitatively into a 250-mL volumetric flask. Rinse the last traces from the boat and the neck of the volumetric flask with a squirt bottle. Add about 150 mL of deionized water, swirl to dissolve completely, carefully dilute to volume, and mix thoroughly.

CALIBRATION OF THE pH METER

A pH meter needs to be calibrated every time it is used for an experiment, and should be checked for accuracy periodically when doing a series of measurements. pH and reference electrodes tend to drift with time owing to changes in temperature, the surfaces of the electrodes, and other factors.

1. Obtain a pH meter, electrode, and buffer solutions from one of the laboratory instructors. Be sure to sign and print your name on the sign in sheet for the pH meter and electrode. Record the instrument numbers found on the outside of the electrode and meter into your lab book. Also, be sure that before you sign for an electrode that the plastic cap on the side of the electrode covering the inlet for the internal filling solution is securely in place. If the cap has been removed notify a TA immediately.

2. Turn on the pH meter and let it warm up for 10-15 minutes. Rinse the combination pH electrode thoroughly with deionized water and gently pat off the excess water with a kimwipe.

3. Insert the electrode into a small beaker (or vial) containing pH 4 buffer solution. Press **CALIBRATE** and wait for **OK**.

4. Rinse the pH electrode with deionized water and insert it into a small beaker with pH 10 buffer. Press **CALIBRATE** again to perform a 2-point calibration. If a high enough % value is not obtained for the slope, an **ERR** message will appear. Repeat the calibration one or two times more. If **ERR** still appears, consult a Teaching Assistant. Record the slope value in your notebook.

5. Rinse the electrode thoroughly and pat dry.

6. When not in use during the experiment, keep the pH electrode immersed in deionized water.

7. When you have completed the entire experiment, the pH electrode should be stored in pH 7 buffer solution.

STANDARDIZATION OF 0.25 M NaOH

Standardize the 0.25 M NaOH solution you prepared by means of a conventional acid-base titration using phenolphthalein indicator.

1. Fill a 50-mL burette in proper manner with the prepared NaOH solution.

2. Pipet 25 mL of the KHP standard solution into a 250-mL Erlenmeyer flask. Add 3 drops of phenolphthalein indicator, and titrate carefully to the first permanent light pink color. (What should the endpoint volume of NaOH be, given the nominal concentrations of the two solutions?)