Preparation and Standardization of Acid and Base Solutions, and Testing of Unknowns

EXPERIMENT 20G

A common laboratory procedure is to determine the concentration of an acid or base solution by titrating it against a solution of known concentration. In an earlier experiment (16B) you were introduced to the method of preparing a standard solution. In a subsequent experiment (20C) you performed an acid-base titration to determine the concentration of acetic acid in vinegar, having been given a sodium hydroxide solution of known molarity. In this experiment you will have to standardize the NaOH yourself.

You will first have to prepare a solution of an acid of known concentration by weighing out a sample and making it up to a known volume in a volumetric flask. To be suitable for such a use, a substance must be very pure and stable. Also, it must not absorb water from the air. A chemical such as this is called a *primary standard*. Sodium hydroxide cannot be used as a primary standard because it is difficult to obtain 100% pure, it readily adsorbs moisture from the air, and it reacts with the carbon dioxide in the air. A common primary standard for acid-base titrations is oxalic acid, which occurs in the crystalline form as the dihydrate $H_2C_2O_4 \cdot 2H_2O$ (or $(COOH)_2 \cdot 2H_2O$). This is the primary standard that you will use in Part I of this experiment. (It must be of analytical reagent purity.)

After preparing the solution of oxalic acid of known molarity, you will carry out a titration with sodium hydroxide solution in order to determine the molarity of the NaOH (Part II). This standardized NaOH can have a variety of uses, such as determining the molar mass of an unknown solid acid (Part III) or the molarity of an unknown acid solution (Part IV). Keep in mind that it is important to make all measurements with as much accuracy as possible.

In this experiment you will also have the opportunity to do some optional procedures which you will design yourself (Parts V and VI).

OBJECTIVES

1. to prepare a standard solution of oxalic acid and use it to standardize an unknown sodium hydroxide solution
2. to determine the molar mass of an unknown solid acid by titration with standardized NaOH solution
3. to determine the pH and molarity of an unknown acid solution and calculate the K_a from the results
4. to analyze a variety of other unknown solutions by titration

MATERIALS

Apparatus

centigram balance
beaker (100 mL)
beaker (250 mL)
funnel
volumetric flask (250 mL)
wash bottle
buret
pipet (25 mL)
suction bulb
pH meter (or universal indicator paper or solution)
stoppered bottle (500 mL)
label
stand
buret clamp
lab apron
safety goggles

Reagents

oxalic acid (crystals)
sodium hydroxide (NaOH) solution (approx. 0.1*M*)
phenolphthalein indicator
unknown solid acid
unknown weak acid solution
Optional Reagents
soft drink
apple, lemon, or grapefruit juice
unknown HCl solution
household ammonia
limewater
bromcresol green indicator
solid sodium carbonate
antacid tablet

PROCEDURE

Part I Preparation of a Primary Standard Acid

CAUTION: Oxalic acid is poisonous. Do not get any in your mouth. Do not swallow any. Always use the suction bulb to withdraw the oxalic acid into the pipet.

1. Before coming to the laboratory, calculate the mass of oxalic acid, $H_2C_2O_4 \cdot 2H_2O$, that you will need to make up 250.0 mL of a 0.0500*M* solution.
2. Put on your lab apron and safety goggles.
3. Accurately determine the mass of an empty (clean and dry) 100 mL beaker and record it in Table 1 in your notebook.
4. Measure into the beaker the amount of oxalic acid that you have calculated you need and accurately determine the mass of the oxalic acid and the beaker. Record this figure in Table 1. Do not spend much time trying to get exactly the same mass as you calculated. The important thing is to record accurately the mass you do have, and to calculate the molarity from this mass. For example, the mass you use may give the solution a molarity of 0.0496*M*. This is perfectly acceptable, provided that you use this figure in your calculations.
6. Dissolve the oxalic acid in water, and pour the solution through a funnel into a 250 mL volumetric flask. Wash the beaker with water twice, and add these washings to the flask. Now add water to the flask until the level is up to the mark. (Use a wash bottle as you get close to the mark.) Stopper the flask, and shake to ensure that the solution is homogeneous. You now have your standard solution of oxalic acid.

Part II Standardization of an Unknown NaOH Solution

1. Obtain a 500 mL bottle with a stopper and fill it with NaOH solution of unknown molarity. Label it with your name and class.

2. Add about 15 mL of the NaOH solution to a buret through a funnel, rinse it back and forth, then discard it through the tip into the sink.

3. Fill up the buret with more NaOH solution and allow some to drain in order to remove any air bubbles in the tip. Remove the funnel.

4. Using the suction bulb on the end of your pipet, withdraw about 5 mL of oxalic acid, rinse it around in the pipet, and discard it. Then withdraw 25 mL of the standard oxalic acid solution and transfer it to a 250 mL Erlenmeyer flask. The correct volume is delivered when you have touched the tip of the pipet to the side of the flask. Do not blow through the pipet. (*Note*: Depending on the shape and size of your pipet and volumetric flask, you may have to transfer the oxalic acid first to a clean, dry beaker since the pipet will not reach deep enough into the volumetric flask.)

5. Add 3 drops of phenolphthalein solution to the acid in the Erlenmeyer flask.

6. Read the initial volume of NaOH in the buret as accurately as you can, and record it in Table 2. Then open the valve on the buret. Allow the NaOH solution to run into the flask and swirl constantly to ensure thorough mixing.

7. After a time, you will notice a pink color that appears where the NaOH enters the liquid in the flask. When this color takes a longer time to disperse and disappear, slow down the rate of addition of NaOH until eventually you are adding it a drop at a time. Stop the titration when the faintest possible pink color stays in the flask for about 20 s. Read the final volume of the NaOH in the buret and record it in Table 2. (The difference between the initial reading and the final reading represents the volume of NaOH required to neutralize the oxalic acid.)

8. If you are at all in doubt as to whether you have a pale pink color, take the reading anyway, then add one more drop. If the color immediately becomes much darker, the reading you took was probably the most accurate result. This is called the *endpoint* of the titration. Discard the solution down the sink.

9. Pipet another 25 mL sample of oxalic acid into the flask and again add 3 drops of phenolphthalein. Refill the buret (if necessary) and repeat the titration. Run in NaOH to within 1 mL of the volume needed in the first titration, then add the solution a drop at a time, swirling after each drop, until you get the faint pink endpoint. Repeat the titration until you have two readings that agree to within 0.1 mL.

10. Store your labelled bottle of standardized NaOH (as instructed by your teacher) until the next laboratory period, when Parts III and IV will be done.

CAUTION: Sodium hydroxide solution is corrosive to skin, eyes, and clothing. When handling NaOH, wear safety goggles, full face shield, gloves, and lab apron. Wash spills and splashes off your skin and clothing immediately using plenty of water. Call your teacher.

CAUTION: Phenolphthalein solution is flammable. Make sure there are no burner flames in the vicinity.

CAUTION: You must assume that any unknowns you are dealing with could be poisonous. Do not get any in your mouth and do not swallow any.

Part III Determination of the Molar Mass of an Unknown Solid Acid

1. Obtain a vial containing an unknown solid acid from your teacher. Record the identifying number or letter in Table 3.

2. Weigh out about 0.75 g of the solid acid into a clean, dry beaker, and record the mass accurately in Table 3. It does not have to be exactly 0.75 g, as long as you know exactly how much you have.

3. Dissolve the acid in about 40 mL of water and transfer the solution to an Erlenmeyer flask. Rinse the beaker twice into the flask to ensure that all the acid solution is transferred. (The amount of water added does not affect the results.) Add 3 drops of phenolphthalein.
4. Run in NaOH from a buret as in Part II, measuring the volume required to reach the endpoint. Record this figure in Table 3.
5. Repeat Steps 2 to 4 until you get two readings in close agreement. (If you did not have exactly the same mass each time, check whether the results agree by determining the ratio of the volumes and comparing it with the ratio of the masses used. Alternatively, follow the calculations set out in the Questions and Calculations section to determine the molar mass of the acid, and see whether those results agree within experimental error.)

Part IV Determination of K_a for an Unknown Monoprotic Weak Acid

1. Obtain approximately 100 mL of the unknown weak acid provided. Record any identifying number or letter in Table 4, if more than one unknown is available.
2. Measure the pH of the solution. The most accurate way of doing this is with a pH meter, if one is available in the lab. If so, your teacher will give instructions on its use and calibration. Otherwise, use universal indicator paper or solution to determine the pH.
3. Use the suction bulb on your pipet to deliver a 25.00 mL portion of the unknown acid into an Erlenmeyer flask. (Rinse the pipet with the acid first.) Add 3 drops of phenolphthalein.
4. Titrate with your standard NaOH solution as you did in Parts II and III, until you get two results agreeing, or until you run out of time.

Analysis: Calculations (including uncertainties) for parts I-N

Evaluation: sources of error
suggested improvements

Conclusion: State your results