Experiment 6

Titration of an Unknown Acid

Prepared by **Stephen E. Schullery** and **Ross Nord**, Eastern Michigan University

PURPOSE

To determine the apparent molar mass of an unknown monoprotic acid by titrating with a standardized NaOH solution.

ACIDS AND BASES

The notion of acids and bases is one of the most widely used principles in chemistry. Because of this variety of applications, several definitions of acid and base have been developed.

Arrhenius: An acid ionizes in water to give a hydrated proton (indicated as H_3O^+ or H^+); a base ionizes in water to give hydroxide ion, OH^-.

Bronsted-Lowry: An acid is a proton donor; a base is a proton acceptor.

Lewis: An acid is an electron-pair acceptor; a base is an electron-pair donor.

In general chemistry, we use a combination of the Bronsted-Lowry and Arrhenius definitions. If you go on to study organic chemistry, you will find the Lewis definition more commonly used.

The reaction between an acid and a base is called **neutralization**. The neutralization of Arrhenius acids and bases results in the formation of a salt and more solvent.

TITRATION

Titration is the process of adding just enough acid to neutralize a sample of base, or vice versa. The solution to be added, usually from a buret, is called the **titrant**. The purpose is usually to determine the concentration (or amount) of whichever reactant was unknown.

Some trick must be used to signal the **equivalence point**, where exactly stoichiometric amounts have been mixed. Chemical indicators, which change color and pH meters are the most common methods.

Titrations aren't just for acids and bases. The technique is applicable to any stoichiometric reaction, e.g., precipitations, oxidation-reduction reactions, and even biological effects.

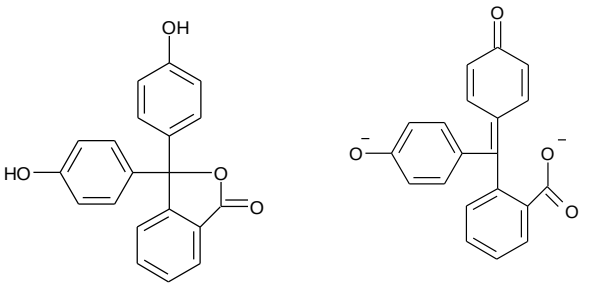
HOW IS THE EQUIVALENCE PT. DETECTED?

The trick is to simultaneously titrate a tiny amount of another acid that changes color when it is converted to its base form. These **indicator** molecules will change the color of the solution at the same point in the titration when the other acid (or base) has been neutralized. Because indicators are so highly colored, only a tiny amount is used during the titration. So, the extra base (or acid) required to titrate the indicator (less than a drop) doesn't affect the result.

The point where the indicator changes color is called the **endpoint** (since the color changes at the end of the titration). Often “endpoint” and “equivalence point” are used interchangeably, but you now know the difference between them.

Numerous "indicator" molecules are known. Many are naturally occurring molecules, such as the pigments in vegetables, berries and flowers. Some flowers even change color depending on whether the soil is acidic or alkaline.

The indicator used in this experiment is phenolphthalein.



Phenolphthalein

acid form (colorless) base form (pink)

The initial solution is acidic, so even after adding a few drops of phenolphthalein, it will be colorless at the start of the titration. When sufficient base is added, the solution will turn a very faint pink at the endpoint. If too much base is added, the solution will turn bright pink. This is called "overshooting" the endpoint.

IN THIS EXPERIMENT

You will determine the molar mass of an unknown solid acid by titrating it with a solution of NaOH(aq) of a known concentration.

PRE-LABORATORY PREPARATION

1. Read the procedure and data analysis sections of the experiment.

2. Complete the computer-generated PRELAB assignment. Refer to the procedure and data analysis sections of the experiment as needed. The prelab questions for this experiment aid in determining the sample size to use, as well as replicating the data analysis.

EXPERIMENTAL SECTION

REAGENTS PROVIDED

Sodium hydroxide, approximately 0.05 *M*.
Phenolphthalein, 1% in ethanol.
Unknown acid, solid.

Hazardous Chemicals

NaOH is very caustic. Do not get it on your clothing, belongings or favorite body parts. Neutralize and clean up any spills immediately. Immediately rinse any spills off your body with lots of water.

WASTE DISPOSAL

All of the chemicals used in this experiment may be safely disposed of by washing down the sink.

TECHNIQUES

USING BURETS

A buret is used to accurately measure the volume of a liquid. It is not quite as accurate as a volumetric pipet, but it is more accurate than graduated cylinders or beakers. It is also very easy to use to deliver arbitrary amounts of liquid.

The scale on a buret is set up to tell you how much solution has been delivered. Thus, zero is at the top of the scale. The volume of solution delivered is determined by subtracting the initial reading from the final reading, so it is essential that both readings be recorded.

Whenever you read calibrated glassware you should try to estimate one decimal place more than the closest calibration marks. So, for a buret with marks every 0.1 mL, you should try and read it to the nearest 0.01 mL.

Because the bottom of the meniscus can be located most precisely, it is used for defining the position of the meniscus. Be sure to have your eye on the same horizontal level as the meniscus, to avoid so-called parallax error. You may need to use a step-stool.

Cleaning a Buret:

1. Rinse the buret with distilled water.

Close the stopcock and fill the buret with distilled water. Drain a few mL of water out through the stopcock. Then close the stopcock, invert the buret (over the sink) and let the water drain out (opening the stopcock again can speed this up).

If liquid will not drain through the open stopcock, get assistance from the instructor.

If drops of water are seen clinging to the *inside* of the buret after the draining is complete, the buret is dirty. Those clinging drops would make a significant error in the results.

2. If the buret is dirty, add about 10 mL of glassware detergent solution (Alconox, found by the front sink), and scrub the inside with a long brush.

3. Rinse three times, with distilled water, to remove the detergent.

Time may be saved on initial rinses by emptying rinses out the top of the buret, but part of the final rinse should be drained through the stopcock.

If there are still clinging drops after the final rinsing, repeat the addition of detergent, scrubbing, and rinsing or get assistance from the instructor.

PROCEDURE

You will do this experiment individually.

PREPARATION OF AN ACID SAMPLE

1. Obtain a test tube containing an unknown acid and record the number.

2. Weigh out the proper amount of your solid unknown acid (see prelab calculation II).

a) Gently, fold a piece of glassine weighing paper in half and then unfold it. This creates a spout to aid in pouring the acid.

b) Place the unfolded weighing paper on the balance and tare it, so it reads 0.000 (± 0.001) g.

c) Use a nickel spatula to slowly add the desired amount of unknown acid. Note that there is no need to weigh out **exactly** the amount of acid calculated, as long as it's approximately the proper amount (± 0.030 g) and you know **exactly** what it is. ***Record the actual mass on your data sheet.***

Clean up any acid that spills onto the balance or the counter.

3. Carefully pour the acid into a 125 mL Erlenmeyer flask, add about 50 mL of distilled water to the flask, and swirl the flask to dissolve the acid.

If necessary, use a clean stirring rod to break up large chunks of solid to speed up the mixing. Do not remove the stirring rod until the titration is complete (since drops of solution cling to the rod and their removal will affect the results).

4. Add two to four drops of phenolphthalein solution to the acid sample.

TITRATION OF AN ACID SAMPLE

5. Clean the 25-mL buret as described in the Techniques section.

It is also a good idea to rotate the stopcock and *gently* tug on it and the glass tip to make sure they do not accidently come out in the middle of the experiment.

6. Rinse the buret with the provided NaOH solution.

(a) Mount the buret in a buret stand. Close the stopcock and, using a funnel, add 5-10 mL of the NaOH(aq).

(b) Remove the buret from the stand. Tilt and roll the buret so that the entire inner surface of the buret is rinsed by the solution.

(c) Drain the rinse solution out through the stopcock and discard this solution.

7. Mount the buret in a buret stand. Using a funnel, fill the buret with the provided NaOH solution. *Record the actual molarity of the solution on your data sheet*.

It is best to fill the buret an inch or two above the zero line and then open the stopcock to drain the excess (into a waste beaker) until the meniscus rests on the zero line. This should flush all air bubbles from the stopcock and delivery tip.

The buret doesn't have to be filled exactly to the zero mark because you are going to be using a difference in readings. However, your arithmetic will be slightly easier if the meniscus starts out exactly on zero.

8. Record the initial reading of the meniscus, estimating to the nearest 0.01 mL.

The number you record is the reading exactly as it appears on the buret. Buret readings correspond to the volume delivered, not the volume still in the buret, so DO NOT subtract what you read from 25!

9. Titrate the acid sample. *Record the final meniscus level in your buret to the nearest 0.01 mL*.

Drain the NaOH into the flask by controlling the stopcock with one hand while gently swirling the flask with your other hand (or stirring with a stirring rod). *Be careful to keep the flask opening directly under the tip of the buret,*

The challenge here is to get to the endpoint reasonably quickly without overshooting it. In the prelab, we calculated the amount of acid to use by assuming we would need 15 mL of base to titrate it. Even though your unknown will likely have a molar mass different from that in the prelab, you can safely add the first 8 mL of base very rapidly.

As you get within a few mL of the endpoint, you will notice the solution begins to turn pink where the NaOH drops hit, but then clears up with continued swirling. As the endpoint nears, add NaOH one drop at a time. Drops can be "split" by rapid rotation of the stopcock plug (which can be hard to do), or by touching a small drop off the buret tip onto the end of a stirring rod, which is immersed into the solution.

The endpoint is signaled when the first very faint trace of pink color persists for about half a minute. Performing the titration over a piece of white paper aids detection of the pink endpoint.

If you overshoot the endpoint, the solution will be more than just slightly pink. Record the color at the endpoint on your data sheet.

10. Prepare and titrate two more acid samples, as above (don't forget the phenolphthalein).

Be sure to thoroughly rinse out the flask with distilled water between trials. However, it does not need to be dried (since more distilled water will be added).

This will go a lot faster because you can take advantage of what you learned from the first titration. Proportionately more or less titrant will be needed for acid samples that are larger or smaller than the first.

You can quickly drain most of the required titrant into the titration flask, then slow down and begin adding dropwise when you know you are getting close (within 1 mL) to the endpoint.

Always record both initial and final meniscus readings to within .01 mL.

11. Decide if you need to do more titrations.

Your grade heavily depends upon the quality of your results. Ideally, you should have three titrations that demonstrate good precision and where you did not significantly overshoot the endpoint. So, do as many titrations as you need to do. However, you have a limited time to complete the lab and should allow yourself about 30 minutes at the end to do the calculations. *If you prepare more samples, be sure to add phenolphathalein*!

A good measure of your precision can be obtained by dividing the volume of NaOH used (in mL) by the mass of acid used (in g). If you have several trials with volume-to-mass ratios within 2 mL/g of each other, then your precision is good.

Rinse your glassware with distilled water. Dry (at least the outside) with a paper towel. Return it to the drawer from whence it came.

Name _____ Station Used _____ Instructor/Day/Time _____

Station Checked & Approved _____

DATA SHEET

Record all data using the appropriate number of significant figures. Notice that the units are included in the column headings so that it is not necessary to record them by each reading in the table. Also, extra rows are provided in case you want to do extra titrations. (Add rows below, or on the back, if necessary.)

Unknown Acid Number: _____

Concentration of NaOH solution: _____

(1) The values in the "Volume of NaOH used" column will be calculated by subtracting the "Initial Buret Reading" from the "Final Buret Reading".

(2) Divide the "Volume of NaOH used" by the "Mass of Acid used" to obtain the "Ratio of Volume to Mass" for each trial. ***Use these values to determine which of your titrations were the most precise. Put stars by these trials and only use them when doing your final calculations.***

Trial Number	Mass of acid used (g)	Initial Buret Reading (mL)	Final Buret Reading (mL)	Color of Endpoint	Calculated Volume of NaOH used (mL)	Calculated Ratio of Volume to Mass (mL/g)
1						
2						
3						

DATA ANALYSIS

All calculations must be clearly organized, make proper use of significant figures and include the units. *Only show the calculation for your first good trial for each type of calculation*.

1. Based upon the volume used and the provided molarity, calculate the moles of NaOH used to titrate each unknown acid sample. Spaces are provided below for up to four good trials.

sample 1: _____

sample 2: _____

sample 3: _____

sample 4: _____

2. Calculate the molar mass of the unknown acid for each good titration sample.

sample 1: _____

sample 2: _____

sample 3: _____

sample 4: _____

3. Calculate the average molar mass of the unknown acid.

average molar mass _____

Unknown Number _____