Standardizing a Sodium Hydroxide Solution

# Introduction

A titration is an analytical procedure used to determine the concentration of a sample by reacting it with a solution of known concentration.  The solution in the *buret*, which is an apparatus used to measure volume accurately, is called the *titrant*. The sample being analyzed is called the *analyte*.

One advantage of a titration is that volume can be measured consistently to four significant figures (to the nearest 0.01 mL) with just the naked eye. If the mass of a sample is measured on an analytical balance to the nearest 0.0001 g, then the molar mass of a compound can be measured accurately to the closest 0.01 g/mol, or the purity of a sample can be known to the closest 0.01%.

This is possible because the *endpoint* of a titration, when the reaction is complete, can be known within a single drop of titrant. An experienced titrator can complete a titration within a half a drop or even a quarter of a drop, where one drop may represent a volume of 0.03 mL or less.

A titration requires an *indicator*, which identifies when the reaction is complete. The type of reaction explored in this experiment is an acid-base reaction. The net ionic equation for an acid-base reaction is

H+ + OH- -> H2O

In simple terms, an acid is a compound with the H+ ion attached; when it is removed, the substance has become a base.

There are many substances that have distinct colors when in the acid or base forms. For example, bromothymol blue is yellow in the acid form but blue in the base form. If a small amount of bromothymol blue is present in solution as an indicator, as soon as the titrant has reacted with all the analyte, it begins to react with the indicator. Since the indicator is present in such a small amount (a drop or two), the conversion from acid to base, or vice versa, occurs with just a single drop of titrant. The indicator for this reaction is phenolphthalein, which is clear in its acid form but pink in its base form.

The measurement of an analyte will be accurate only if the concentration of the titrant is known accurately. Sometimes, a titrant can be prepared by measuring its mass as a dry sample to four or five significant figures and dissolving in glassware with an accuracy of four or five significant figures. In that case, the titrant is called a *primary standard*. But this is not always possible.

A common titrant for an acid-base titration is sodium hydroxide, NaOH. Sodium hydroxide readily absorbs water from the air, so its measured mass on an electronic balance is not all sodium hydroxide. Furthermore, aqueous solutions absorb carbon dioxide from the air, which forms an acid in solution and reacts with the sodium hydroxide in solution.

Therefore, the first step of a titration with sodium hydroxide is to *standardize* the solution. We do this by using a primary standard as an analyte. In this experiment, the analyte is the primary standard, oxalic acid. The acid is diprotic, meaning that it has two acid hydrogen atoms:

2 NaOH + 1 H2C2O4 -> 1 Na2C2O4 + 2 H2O

Furthermore, the crystalline form of oxalic acid is a *dihydrate*, meaning for every mole of acid there are two moles of water in the crystal. That is why we use a molar mass of 126.23 g/mole instead of 90/04 g/mole in the calculations.

Titrations are still an important and useful analytical technique in the chemistry laboratory; therefore, it is important for every student enrolled in a general chemistry class to have an opportunity to learn about and practice this experimental technique.

# Materials and Methods

* Oxalic acid dihydrate - Corrosive
* Phenolphthalein indicator – Flammable, Carcinogenic
* 0.1 M NaOH – Corrosive, Mutagenic, Toxic
* Baking soda
* buret
* 250 mL Erlenmeyer flask
* LabQuest and pH probe

# Procedure

## Set Up and Preparation

1. Titrations are usually performed standing, not sitting. If the buret is too high to read correctly, do not stand or kneel on a stool. Instead, step stools are available in the room to see the meniscus at eye level. You can also lower the buret in its holder, or remove it completely from the stand, to obtain an accurate reading.

2. Move all books, backpacks, and papers away from the workspace, except the Report Sheet. Place the buret stand in a location where you feel comfortable in front of it. (The buret stand is initially set toward the back of the table to provide benchtop space before beginning the experiment: it is not necessary to leave it in that position.)

3. Remove the buret from the clamp and observe its markings: 0.00 is at the top of the buret and 50.00 is at the bottom. Replace the buret in the clamp, either on the left-hand side or the right-hand side, depending on which hand you will be using to control the stopcock. The stopcock can also be rotated in the buret, as needed. Make sure the stopcock is in the ‘off’ position: rotated 90 degrees to the buret.

4. Add some water to a 250-mL beaker for practicing titration techniques. Place a waste beaker under the buret. With a buret funnel in one hand and the beaker in the other, raise both to the top of the buret. Put the funnel in the buret and pour the water into the buret. Pour slowly so that the water does not over-fill the buret. Fill the buret close to the zero-mark: between 0.0 and 5.0 mL. Remove the buret funnel and lower both the funnel and the beaker at the same time. (This technique should lessen the chance that you leave the funnel in the buret while titrating, which you should never do.)

5. Slowly turn the stopcock until the titrant fills the tip, eliminating air bubble in the tip. Drain water, as necessary, to ensure that the meniscus is below the 0.0 mark.

6. Read the buret. Have the meniscus at eye level, raising or lowering the buret in the clamp to make that possible. Hold your gloved hand behind the buret to see the white marking more clearly. *Estimate* the volume reading of the titrant to the closest 0.01 mL: imagine ten subdivisions between the marks. Note that the smaller number is on top.

For example: The meniscus is between 3 and 4, so it is 3.*something*. The meniscus is between the third and fourth lines, counting down, so the reading is 3.3*something*. The line appears half-way between the marks, but maybe lower, so I estimate the reading to be 3.36 mL.

## Titration Practice

1. Hold an Erlenmeyer flask loosely by the rim and begin swirling it. Try swirling it with your other hand as well. You will find one hand to be more comfortable than the other: you are either a right-handed or left-handed titrator. (If, during the titration, you find that you are setting down the flask to turn the stopcock, you are swirling the flask with the wrong hand.)

2. Hold the flask under the buret and turn the stopcock so that the titrant is entering the flask full stream. Swirl the flask the whole time and do not let go of the stopcock. Back off the stopcock so the flow rate is not so fast, still swirling the flask. Back off the stopcock so that the titrant is a stream of drops, still swirling. Back off the stopcock, never letting it go, until drops are falling about one per second. Cut the flow rate in half while still swirling. Make the drops fall every 2-3 seconds. Make the drops fall every 5-10 seconds. Control the stopcock so that you can deliberately add one drop at a time, cutting off the flow in between drops.

Refill the buret and practice, as needed, until you feel you can control the flow rate from the buret.

3. Practice reading the buret again.

## A First Titration

1. Empty the flask of tap water.

2. Measure approximately 0.19 g of oxalic acid in a blue weighing boat, measured to the nearest 0.001 g, and record the value on the Report Sheet.

3. Pour the acid into the flask and use a wash bottle to rinse any acid remaining in the weighing boat into the flask.

4. Add around 25 mL of *deionized* water from a wash bottle into the flask: just enough so that the solution will swirl nicely.

5. Add 2-3 drops of phenolphthalein indicator to the flask and set it to the side (not under the buret!)

6. Drain the water from the buret and discard it. Obtain sodium hydroxide in a beaker, either for yourself or enough to share with our neighbors. Fill the buret with the stock solution and drain the air bubble from the tip.

7. Take an initial buret reading and record the value on the Report Sheet. *Only then* will you place the flask under the buret. The flask will remain under the buret until after the second reading of the buret.

8. Begin adding titrant at a fast rate while swirling. If the color remains or expands for more than two seconds, slow down the titration rate. Over time, you should slow down to drops, then fewer drops, until you are adding a drop at a time, just like in the titration practice. Be ready to stop the titrant flow completely to make sure the solution clears, if needed, before starting to titrate again.

9. This is the ***only*** condition for a ‘good’ titration: you add a drop of titrant, swirl, and the pink color disappears. You add the next drop of titrant, swirl, and the color remains pink for at least 15 seconds. (Because of carbon dioxide in the air, the color will fade over time, or if you keep swirling.) The shade of pink does not determine a good titration. If you cannot attest that the last drop you added completed the titration, then it is not a good titration.

10. When the titration is completed, take a final reading of the buret and record the value on the Report Sheet.