

Titration Learning Lab

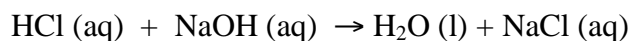
Discussion

Titration experiments quantify how much of a substance is in an “unknown” solution. It is a standard analytical-lab experiment. A *different type of experiment* is used to determine whether or not lead is present in paint, or if some other toxic substance is in the soil, or in ground water. Once the substance is detected, then the question becomes “how much of the substance is in the soil sample or in the water sample?” This is the question that a titration experiment can answer.

A titration experiment uses 1) a known chemical reaction, and 2) an indicator to determine the amount (in grams or in units of concentration (moles/Liters = molarity) of the substance in the sample being tested. The sample being tested is the “unknown”.

Procedure

In this experiment, we will confirm that the (approximately) 0.085 M aqueous NaOH solution that you made in lab, is in fact, 0.085 M NaOH (or whatever number you eventually calculated). In the process, you will (hopefully) see that stoichiometry, and this particular application of it, actually works. The chemical reaction we will use is an example of an acid/base reaction.



The first thing that we will do is clean the glassware that we will use. Some of you noticed that droplets of water sometimes cling to the top of the volumetric flask, or the pipet. Water should not do that. If you notice that water droplets cling to your flask, pipet, or buret, it should be cleaned with soap or other cleaner and distilled water. We will have some long pipet and buret brushes available on a cart for that.

If the distilled water drains freely, and doesn't cling to the sides of the glassware, then you can assume that your pipet or buret is clean, but they are not yet ready to be used. First, they must be rinsed, 3 times, with whatever solution you are going to put into the buret and the pipet. This is to ensure that the concentration of the original solution is not changed (diluted) by the water inside the buret or pipet. Note: the fact that the water droplets do not cling, shows only that the inside of the buret or pipet is not oily (dirty), but the inside glass walls are not *dry*.

Obtain 75- 80 mLs of HCl solution in a clean, dry beaker. Use this solution to rinse the buret 3x, and then fill the buret. You only need about 5 mLs of HCl solution per rinse, and this will be demonstrated in class. Also, note the concentration of the HCl solution and record it in your notebook.

Once cleaned, and rinsed with HCl, fill the buret up to or below the 0.00 mL line with the HCl solution. The hydrochloric acid solution is in a carboy (large plastic container) at the instructor's desk. Using the funnel for the buret, fill the buret.

Please make sure that the spigot is completely closed before walking away with your HCl solution or your distilled water. As a way to remind you of this, turn the spigot up after you are finished.

Use the same cleaning and rinsing procedure to prepare your pipet for use. Your diluted, approx. 0.085M NaOH solution, will be the Unknown or test sample for this experiment. Pour some of your NaOH solution into a small, clean and dry beaker. Attach the suction bulb to your 25 mL pipet, and take up about 3-4 mls of NaOH and rinse the pipet 3x. This will be demonstrated in class. Next, make the “unknown solution” for the experiment by adding 20 mL NaOH, about 25 mL of DI Water (which does not need to be carefully measured, use your graduated cylinder) and 2-3 drops of phenolphthalein indicator solution into a clean 250 mL erlenmeyer flask. Swirl the flask to mix the contents. **USE THE PIPET TO CAREFULLY MEASURE** (read below) **AND RECORD** the amount of NaOH added to the sample. This quantity is very important. Use the appropriate number of significant figures. Do not write “20 mL”, which has only 1 significant figure. When all three substances are mixed, your first sample is ready for the titration experiment.

Reading the buret and the pipet

To start the titration experiment, first record the initial volume of HCl in the buret. We will want to know, in the end, how much of the HCl solution we have added to the solution in the 250 mL erlenmeyer flask, so read the buret down (the way you read a pipet). Do not read “up” from 50.00 mL or subtract the number you obtain from 50.00 mL. Think of it this way: initially, we have added 0.00mL of solution to the erlenmeyer flask. If you filled the buret to a point below the 0.00 line, then record that number (0.08 mL, or 0.16mL or whatever)...this initial reading does *not say* that you have added this amount to the Erlenmeyer flask, it is merely the starting point. At the end of the experiment, you may be at 25.27mL, for example. Then to determine the correct volume added to the erlenmeyer flask, subtract the initial reading from the final reading: $25.27\text{mL} - 0.16\text{mL} = 25.11\text{mL}$, which is the volume of HCl added to the erlenmeyer flask.

The term “titrate” means adding the solution in the buret to the unknown solution in the Erlenmeyer flask. Drop by drop, add the HCl solution to the NaOH solution, while swirling the solution in the Erlenmeyer flask. The reaction states the products: sodium chloride dissolved in water. We know the color of that solution: colorless and clear; however, we have added an indicator to the NaOH solution. The indicator turns a bright color under basic conditions, this indicator turns color around pH 8-10. Below pH 8, the indicator color fades completely to colorless. When the indicator fades completely to colorless, even after one swirl and remains colorless for 30 seconds, we will assume the reaction is completely over. **RECORD THIS FINAL VOLUME IN YOUR NOTEBOOK.** This is the important part for the stoichiometry. We want to end the experiment as soon as we have evidence that the reaction is complete. One single drop will make the difference here, and the volume of one single drop is approximately 0.01-0.04mL. The point at which the indicator *indicates to us* that the experiment is over (in this case when the color change persists for 30 seconds) is called the “endpoint” of the reaction.

Make two more samples and titrate them as well, so that you have a total of 3 trials. You have enough sample for 4 trials. If you know you made an error in one of your first three trials, and if you have time, you should try a fourth trial.

You do NOT need to clean the glassware in between the samples, but you must clean the glassware after the titrations are complete. You only need to clean the glassware with detergent if the solution is not draining cleanly; otherwise, drain and dispose of any excess solution as directed by the instructor, and rinse pipets, burets, and the volumetric flasks with DI WATER 3x.

Then, fill the buret above the 0.00mL line with DI WATER, cork the top, and return to the cart. Also fill the volumetric flask with DI WATER, above the etched line on the neck of the flask. Let the inside of the pipet air dry. Clean the rest of your glassware with soap and water. You can save time and paper towels by rinsing with DI water at the end, setting the glassware upside down in your lockers, and letting the glassware air dry. Calculations are described in the last third of this page.

From the previous lab, as a reminder:

Procedure - measuring liquids

When making measurements in lab, always read “between the lines”. This means that if your pipet has marks to the 0.1 mL, then you should estimate the volume to the hundredths place. That is, if the bottom of the meniscus falls between two lines, estimate whether the level is half way between, a little less than half way, or a little more than half way between the two lines and assign a number to the estimate.

For example: if the meniscus falls between 2.3 – 2.4 mLs and you see the meniscus just below the 2.3 line, then you should estimate that the measurement is 2.31mL or 2.32mL or 2.33 mLs.

If the meniscus is sitting “exactly**” on the line, then you are still estimating to the same decimal place as you were when the meniscus falls between two lines.

For example: if the meniscus falls on the 2.3 mL line, then you should write the measurement as 2.30 mL because you could distinguish it from 2.29 mL and 2.31 mL.

Calculations

The objective of this experiment is learn the technique of a titration experiment, reach the endpoint accurately, and then using stoichiometry, to determine the concentration of NaOH in our “unknown” sample.

What is the unit of the chemical reaction? That is where we need to go, because we are going to use the chemical reaction to relate the amount of HCl in the experiment to the amount of NaOH in the unknown sample.

Use the Molarity of the HCl (a given quantity) and the volume of HCl used in the experiment, to find moles of HCl. Use the mole ratio of HCl to NaOH to determine the number of moles of NaOH that were in the unknown solution, and use the volume of the NaOH solution pipetted into the Erlenmeyer flask to finally obtain the Molarity (moles/L) of NaOH. This number should be within 5% of the number you wrote on your solution label. We will go over this calculation in class. You do not have to finish the calculation before you leave lab, (but you can if you want to!)...We will go over the calculation during lecture, and determine the percent error, and discuss the sources of error in the experiment.