

CHM 115 Lab 3

Titration: Standardize NaOH/Determine impure KHP

Purpose: You will precisely measure the concentration of the NaOH solution you prepared last week by carrying out a series of acid/base neutralizations.

Background: A **neutralization** is when an acid and a base react together to form a salt. You will react a strong base, sodium hydroxide, with a weak acid, potassium hydrogen phthalate (Figure 1). Because potassium hydrogen phthalate is awkward to say/write, abbreviate the name KHP. This is confusing because it looks like a chemical formula, but it's not. There is no phosphorus (P), and remember that potassium's symbol is K, not P. The products formed are KNaP (potassium sodium phthalate) and water.

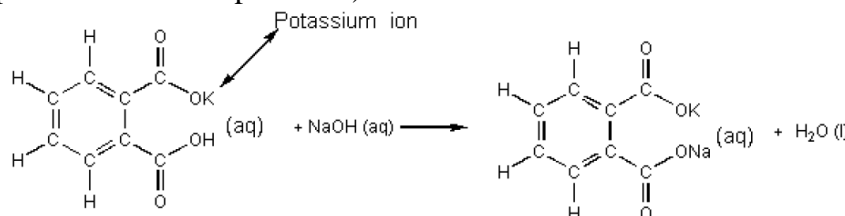


Figure 1. Neutralization reaction of potassium hydrogen phthalate with sodium hydroxide forming sodium potassium phthalate and water.

When you want to know exactly how much of a substance you have, you can carry out a titration. Using an exact amount of one reactant (the **titrant**) you can figure out how much of the other reactant (the **analyte**) must be

present. There has to be some obvious sign when the mole ratio of titrant and analyte match the balanced equation ratio. This is the **equivalence point**. We measure the equivalence point by adding an **indicator** molecule that changes color. The human eye is much better at telling the difference between "clear" and "colored" than it is at distinguishing between two different shades, so the endpoint (when you should stop) is when the solution is the faintest shade of color that you can see (Fig 2). At this point, the balanced equation tells you the ratio of reactants, neither one is in excess. Titrations are very useful in medicine (determining out how much medication someone needs) food processing (figuring out how much fat is in a product) and industry (measuring how much heavy metal is in a waste stream).

For the NaOH solution you made last week, only the approximate concentration is known. You did not use exact volumes (the bottle used is not an exact volumetric piece of glassware) and the deionized water you used contains some impurities (like carbon dioxide). You will measure the exact concentration of NaOH using pure KHP as the titrant. Next, you will turn around and use the exact concentration of this standardized solution. Look at the tutorial videos for titration tips <http://bit.ly/9i8Rn9>



Fig 2 – Colors observed during a titration – don't go too far.

Start

Endpoint

Too Far

Procedure (This is the part you have to write in your notebook):

You will work alone for this lab.

1. Carefully obtain from the desiccator one of the vials of KHP. The molecular weight of KHP is 204.23 g/mol. Make sure your buret is clean and the stopcock is tightened. Rinse the buret three times with 5 mL portions of your NaOH solution. Get any bubbles out of the tip.
2. Fill the buret and adjust the volume between 0 and 1 mL. Read and record the initial volume to two decimal places. Make sure your eye is lined up to avoid parallax errors.
3. Use the analytical balance to weigh at least 1.0 but not more than 1.1 g of KHP. Record all 4 decimal places. Transfer KHP to a 250 mL Erlenmeyer flask, and use deionized water from your squirt bottle to rinse any remaining weigh boat into the flask.
4. Dissolve the KHP in 75 mL of deionized water. Don't worry if not all of it dissolves, just write it down as an observation and make sure it all dissolves before the endpoint. Add 3 drops of phenolphthalein indicator to the flask.
5. Dispense NaOH from the buret into the flask and swirl. When the solution turns faint pink and the color lasts 10 seconds, you have reached the endpoint. It should take 20 - 40 mL of NaOH. Write the final volume down to two decimal places. Expert titrators can split drops to get exactly the endpoint. If it turns dark pink (fuschia) you have overshoot the endpoint. After the titration, the solution may be rinsed down the drain.
6. Repeat the titration 3 times. Keep the mass of KHP used as constant as possible. Calculate the concentration of your NaOH solution and the average deviation (as in lab 1). If the average deviation is less than 0.0002 M, you can stop. If not, do another trial or two.
7. Before the buret is stored, rinse it thoroughly with water, and loosen the stopcock. Put the KHP vial in the box - **not** back in the desiccators. Put the bottle of NaOH back for next week.

| | Trial1 | Trial 2 | Trial 3 | Trial 4 | Trial 5 |
|-------------------|--------|---------|---------|---------|---------|
| Mass KHP | | | | | |
| Initial buret vol | | | | | |
| Final buret vol | | | | | |

Titration 2: Impure KHP/NaOH

Purpose: Using the NaOH you standardized, you will determine how much KHP is in an impure sample.

Background: You will repeat exactly the same reaction, the neutralization of an acid (potassium hydrogen phthalate KHP) with a base (NaOH). This time NaOH will be your titrant, because you

know its exact concentration. The KHP will be the analyte because it is impure, so the amount you actually have is unknown.

Procedure (This is the part you have to write in your notebook):

Locate your brown bottle with the standardized NaOH. It should be mostly full (~ 800 mL). You will be working alone for this lab.

1. Obtain a numbered mixture containing an unknown amount of KHP from the desiccator. Record the unknown number. Rinse out the buret with your standard NaOH solution and set it up for titrating.
2. Weigh to four places past the decimal *one sample* of about a gram; dissolve it in about 75 mL of deionized water; add 3 drops phenolphthalein indicator and titrate to the endpoint with your NaOH solution.
3. For best results, a titration should require 20-40 mL of titrant. If your mixture has very little KHP in it, you may need to use up to 2.0 g of sample, so that more titrant will be required. Do not use more than 2.0 g or we will run out. Do at least three titrations with the same size sample.
4. Calculate how many moles of KHP are present in your sample. Remember you cannot use the molar mass because it is a mixture, not pure KHP. So you'll need to use the concentration of your NaOH that you measured last week to do this calculation. Then find the grams of KHP in each run. Divide the g KHP by the mass of the mixture to find the percent KHP for each run. Find the average % KHP, deviation and average deviation. If your deviation is more than 0.5%, perform more titrations.
5. When you are finished remember to rinse the buret thoroughly with water, loosen the stopcock and return the buret to the rack. Dispose of the remaining NaOH solution, rinse the brown bottle and leave it on the shelf.

| | Trial 1 | Trial 2 | Trial 3 | Trial 4 | Trial 5 |
|-------------------|---------|---------|---------|---------|---------|
| Mass impure KHP | | | | | |
| Initial buret vol | | | | | |
| Final buret vol | | | | | |

Tips:

- Do not throw the data out if you overshoot. One extra drop can cause a huge change in color but a pretty small effect on concentration. Just record it as an observation.
- Use the deionized water squirt bottle to rinse down the sides of your flask if anything splashes.

- Do the first run quickly to get a good idea of where the endpoint will be. Then use very similar masses of KHP for the subsequent runs, so you'll know exactly when to slow down.
- If the volume required to titrate 1 g of KHP is not in the range of 20-50 mL of NaOH, your solution was made wrong (you probably used KOH or measured volume incorrectly). Talk to your instructor.
- Watch significant figures. Rounding errors can affect your precision.
- If you've added more base than should be required and there's still no hint of pink, make sure you didn't forget the phenolphthalein indicator.
- If you're not sure if something is pink, compare it to a white sheet of paper near natural light.