

# ACID-BASE TITRATIONS 1:

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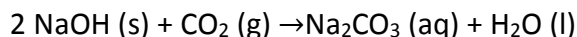
## Standardization of NaOH and Titration of an Unknown Weak Acid

*You should review the section in your textbook that describes proper technique for analytical weighing and for titration. As is usually the case, there are subtleties and pitfalls for the uninformed.*

In this experiment, you will use indicator-based titrations to standardize the stock NaOH solution that your section will be using this term. There is a bit of work required and you probably would rather not go through this procedure again so try to be judicious in your use of the stock solutions and make sure that the carboys are completely off after you are through using them. In the last part of the experiment, you will use the standardized base in the titration of an unknown acid.

## Standard NaOH Solution

Solid sodium hydroxide is hygroscopic, which means that it absorbs moisture from the atmosphere. Once it has a little moisture it also absorbs carbon dioxide which is always present in air. The reaction is:



This means that solid reagent grade sodium hydroxide is not pure enough to weigh and use directly. Furthermore, the carbonate ion interferes in acid-base titrations because 1) it is a weak base, and 2) it tends to make the color change at the end point less sharp. This reaction also takes place in the aqueous phase, where sodium hydroxide in solution reacts with CO<sub>2</sub> from the air to form sodium carbonate. This can change the concentration of standard solutions if steps are not taken to minimize the carbon dioxide uptake. (Note 1) It is therefore necessary to prepare sodium hydroxide solutions in such a way that they have as little carbonate as possible. The most convenient method takes advantage of the fact that sodium carbonate is insoluble in 50 % NaOH solution. Carbonate-free solutions can be obtained simply by diluting 50 % NaOH at the time of use. (Note 2) Other methods are discussed in the textbook.

The concentration of the 1 M NaOH solution provided to you will not be known accurately, so you will measure the concentration of a diluted solution by using it to titrate a well-known amount of acid. This is called **standardization** of the solution. For the titrations, you will need to prepare some 0.1 M NaOH using your calibrated volumetric pipet to transfer 25 mL of the nominally 1.0 M NaOH from your section's carboy to the calibrated 250 mL volumetric flask and then dilute to the mark with the deionized water. Follow the standard procedure for mixing

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(invert the volumetric flask 20 times, allowing the bubble to run all the way up the neck to the stopper) and then transfer the NaOH to your brown plastic bottle and cap it tightly. After standardizing the dilute solution during this experiment, you will back-calculate the concentration of the standardized 1 M solution in the carboy (it will be about 10 times more concentrated).

### Standardization of Sodium Hydroxide Solution

Primary standard potassium hydrogen phthalate will be used to standardize the sodium hydroxide. This monoprotic acid is a crystalline solid and must be dried for 2 hours at 110°C to remove adsorbed water. (Note 3) In order to keep it dry it will be stored in a desiccator until used. (Note 4) You should take your desiccator and vial to the oven, dispense the solid into your vial using the appropriate amount as demonstrated by your TA, put the lid on the desiccator, and return the solid to the oven, all in as expeditious a manner as possible. The solid should not be left out of the oven for more than a minute or two.

**Pre-Calculation:** \*Calculate the weight of primary standard potassium hydrogen phthalate (KHP =  $\text{KHC}_8\text{H}_4\text{O}_4$ , MW= 204.2 g/mol) required to give a 25 mL titration using 0.10 M NaOH.

*\*Pre-calculations are done to help plan the experiment, even when experimental design is not "your job" per se – they help you understand how the experiment works and why the specified amounts are used. They are not part of calculations that are used to determine the final results for the experiment. You will see them in most of the experiments.*

Using the analytical balance weigh out three samples of primary standard potassium hydrogen phthalate into clean 250-mL Erlenmeyer flasks. *Make sure that you close the doors of the balance for the final mass measurement.* Add about 35 mL of DI water to dissolve the solid by swirling.

Test your buret to make sure that it runs clean. Rinse out your buret and its tip three times with small portions of your NaOH solution. (Note 5) Fill the buret and make sure there is no bubble of air under the stopcock. Set the initial buret reading to between 0 and 5 mL and read the initial volume to 0.01 mL. (Note 6) *Using the buret well in a titration is another manual skill that requires some practice to master. You should be able to obtain a single drop of titrant with your stopcock before you attempt a titration in this experiment (being careful about your use of time and reagents).*

Add 5 drops of 0.2 % phenolphthalein indicator solution to the first flask of potassium hydrogen phthalate solution and titrate. Just before reaching the endpoint, rinse the inside wall of the

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flask with a stream of DI water from your wash bottle. The end point is the first faint pink color that persists for 20 – 30 seconds. (Note 7) Read the buret to the nearest 0.01 mL.

Titrate the remaining solutions. Calculate the molarity of the stock NaOH solution (using the factor of "10" that applies to your glassware), the standard deviation of your three results and the relative standard deviation. If you have done careful work your relative standard deviation should be less than 5 parts per thousand. Otherwise you should consult your TA and potentially, do more trials.

### Titration of an Unknown Acid

Obtain about 150 mL of unknown acid in a clean dry beaker. Rinse your 25-mL pipet with three small portions of this solution and then pipet 25.00 mL into a 250-mL Erlenmeyer flask. Add 5 drops of phenolphthalein indicator solution and titrate with your standard 0.1 M NaOH solution. Repeat the titration until you are confident that you have three good results. Calculate the average molarity of the acid, and the standard deviation and relative standard deviation (in ppt) of your replicate results. Note that this solution was not diluted, so the "factor of 10" is not part of this calculation, but the appropriate base concentration is the diluted (not stock) version obtained during the standardization.

### REQUIRED MEASUREMENTS

The PreLab calculation spreadsheet – with correct answers – must be submitted before you can begin work on the experiment. At the end of the experiment, you must present YOUR results for the standardization of the NaOH and for the titrations of the unknown acid to your lab TA (before the last ten minutes of lab, so you have time to clean up and leave on time). As usual, results include the mean and relative standard deviation (‰) of your replicate determinations of the concentrations of the two solutions.

Results Table

|              | Concentration (Carboy) | Stdev | RSD (‰) |
|--------------|------------------------|-------|---------|
| NaOH         |                        |       |         |
| Unknown Acid |                        |       |         |

### NOTES

(1) The water used to make standard basic solutions must be carbonate free. If there is any doubt it should be boiled before use in order to drive off dissolved carbon dioxide. Portland tap water is virtually mineral carbonate free and after distillation it can be used directly. In certain geographical areas, however, the water contains substantial carbonate and although it may not

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be completely removed by distillation it can be eliminated by boiling. Deionized water is generally carbonate-free.

(2) 50 % NaOH solution frequently contains pieces of insoluble sodium carbonate. Usually these can be avoided or fished out but if this is not possible it is necessary to filter the solution through a wad of glass wool.

(3) Standard operating procedure is to dry for one hour at 110 °C. However, in a large class there is so much opening and closing of the oven that the temperature cannot be maintained for the shorter period of time. If the oven is not busy then one hour drying is sufficient. (Check with your TA because this usually has been done already.)

(4) Check your desiccator to be sure it has good desiccant in the bottom (it should be blue, not red) and that the lid seals.

(5) Use a small portion of the 0.1 M solution to rinse your funnel into a waste beaker to avoid contamination; then put a few mL into the buret and hold the buret nearly horizontal while rotating with the top in your waste beaker to coat the inside of the buret; after a couple of these rinses, let the last rinse run through the tip and stopcock; BUT don't overuse the solution, otherwise you may run out for the titration.

(6) If the liquid level inside the buret is changed rapidly allow a minute for drainage of the upper wall before making a reading.

(7) Absorption of atmospheric carbon dioxide causes the color to fade over several minutes time.

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