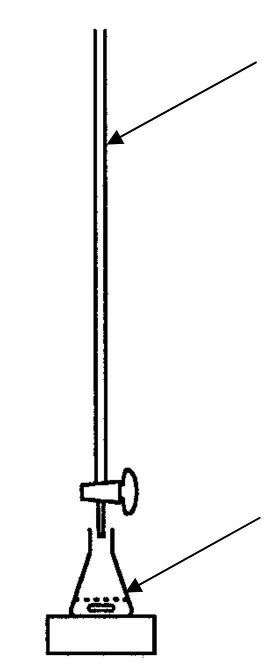
Lab 6: Titrations

Standardization of NaOH and Determination of Acetic Acid Concentration

Introduction

In the Stoichiometry I lab, the equivalence point of the bleach-hydrogen peroxide reaction was determined by measuring the limiting volume of O_2 (g) produced. In the Fe(II)-phen reaction, the limiting absorbance of the $Fe(phen)_n^{2+}$ product was measured. In the bleach-peroxide reaction, because the experimental setup did not allow for continuous addition of one reagent to another, we had to make a number of separate runs. In the Fe(II)-phen reaction we were able to make 100 µL additions of Fe(II) to the same solution of phen in a procedure that allowed us greater speed and accuracy. As the Fe(II)-phen reaction indicates, it is possible to make continuous and controlled addition of one reagent to another as long as: (i) the reaction is fast, (ii) the reaction is of fixed stoichiometry, and (iii) there exists a procedure via which the equivalence point can be identified.

In this experiment, the equivalence point will be identified by adding an acid/base indicator that changes color over a specific range of pH (acid strength). An efficient way to deliver controlled volumes of one solution or liquid to another is to use a buret in a procedure called a **titration**.

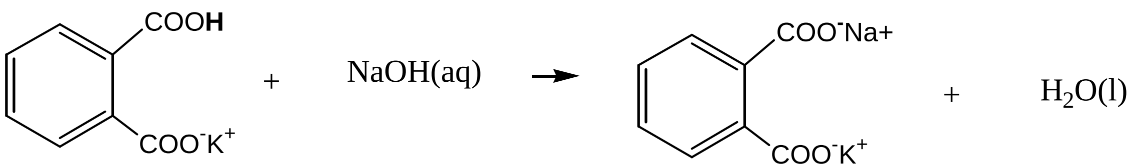


A known volume of titrant is carefully delivered from the buret. As the equivalence point is approached, slow additions are necessary to avoid overshooting the equivalence point. You should be able to identify the endpoint to within a drop (~0.05 mL) or fraction thereof. A carefully designed titration should yield at least 3 significant figures in the volume delivered.

Moles of reagent to be titrated. (If a known mass of reagent is added, moles = grams/molar mass, regardless of the volume of water the solid is dissolved in.)

In this experiment, you will perform two titrations. The first will allow you to accurately determine the concentration of the ~0.1 M NaOH solution to at least three significant figures.. You will then use that solution to titrate an unknown solution of acetic acid (CH_3COOH) and determine its concentration.

When NaOH(s) is purchased from a supplier, it does not come in adequate purity. In addition, NaOH is hygroscopic, meaning that it absorbs water and carbon dioxide from the air. Therefore, a NaOH solution needs to be standardized to determine its exact concentration before it can be used to titrate an unknown solution. For the standardization of NaOH(aq) in this experiment, potassium hydrogen phthalate, abbreviated KHP (204.22 g/mole), is used. KHP is a weak acid with one ionizable proton (shown in bold print in the equation below). KHP is also a primary standard, meaning that it is available from a supplier with greater than 99% purity.



You will weigh out an amount of KHP so that you know the exact mass (and, therefore, moles) of the sample and dissolve it in DI water in an Erlenmeyer flask. After adding a couple drops of the endpoint indicator you will titrate this KHP solution with the NaOH(aq) that is approximately 0.1 M. At the equivalence point, you will have added a number of moles of NaOH to react exactly with all of the moles of KHP so that neither reagent is in excess. Knowing the moles of NaOH added and the volume delivered from the buret, you can calculate the exact concentration of the NaOH solution.

The standardized NaOH will then be used to titrate an unknown acetic acid (CH_3COOH) solution with the goal of determining its exact concentration. You will deliver an exact volume of the acetic acid solution to an Erlenmeyer flask. As with the standardization titration, phenolphthalein will be used as the endpoint indicator. With the exact molarity of the NaOH and the volume delivered from the buret known, together with the volume of the acetic acid solution, the concentration of the acetic acid solution can be calculated.

The prelab assignment on WebAssign addresses the following:

- When titrating a known mass of KHP with a NaOH solution expected to be 0.1 M, what volume of NaOH would you expect to add in order to reach the equivalence point? (This is a good way to estimate where your endpoint will be for the titration so that you know when to start adding the NaOH drop-wise, in this case a few milliliters before the calculated endpoint.)

- Provided with data similar to what you will collect in lab: volume of unknown acetic acid solution, concentration of NaOH solution, and initial and final buret volume readings, calculate the moles of NaOH delivered from the buret to reach the equivalence point and determine the concentration of the acetic acid solution.

Helpful information

- The basics of acid-base reactions and titrations are covered in Zumdahl Section 4.9. Acids and bases are covered more extensively in chapter 7 and titrations in Section 8.5.
- In general, an acid is a proton donor and a base is a proton acceptor. The net ionic equation for a neutralization reaction is $H^+_{(aq)} + OH^-_{(aq)} \rightarrow H_2O_{(l)}$
- A titration is a form of volumetric analysis that uses one solution to analyze another. Generally, the number of moles of a reagent added from a buret is used to determine the number of moles of the other reagent present in the flask at the start of the reaction.
- If the reaction stoichiometry for a titration is 1:1, as it is in this case, at the equivalence point the moles of reagent delivered from the buret will equal the moles of the reagent in the flask, such that $M_bL_b = M_fL_f$, where M is molarity and L is the volume in liters. If three of the four parameters are known the fourth can be calculated.
- *Equivalence point*: point at which the moles of titrant added react exactly with the substance being determined; *Endpoint*: point at which the indicator changes color; Ideally, an indicator is chosen so that the endpoint matches the equivalence point as closely as possible – phenolphthalein satisfies this need for this experiment.
- Practical tips:
    - Use a clean buret. Rinse well with DI water and small portions of the solution it will be holding before filling it to perform the titration.
    - You do not need to start with the buret filled to the 0.00 mL mark (at the top), but you need to know exactly what the starting volume is.
    - Use small additions as you approach the endpoint (the pink color will begin to linger longer before disappearing). You should be able to be within one drop (0.05 mL) of the correct endpoint.
    - Read the *bottom* of the meniscus (see pictures in “Demonstration” link).
    - Avoid parallax error - your eye must be at same height as the meniscus in order to get an accurate reading.

Safety Considerations

- NaOH is a strong base and a corrosive chemical. Use caution when handling this reagent. If you get some base on you, wash areas of contact with large amounts of water.
- Make sure that the nut on the stopcock is tight and that liquid in the buret does not leak out before you fill it with the NaOH. You can check this when you are rinsing it with the DI water.