ACID-BASE TITRATIONS 1:

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 little bit of work required and you probably would rather not go through this procedure again in two weeks 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.* You will then use the standardized base in the titration of an unknown acid.

Standard Sodium Hydroxide 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:

$$2\ NaOH\ (s) + CO_2\ (g) \rightarrow Na_2CO_3\ (aq) + H_2O\ (l)$$

Therefore solid reagent grade sodium hydroxide is not pure enough to weigh directly. Furthermore, the carbonate ion interferes in acid-base titrations because 1) it is a 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 is converted to 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 are free of carbonate impurity. 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. (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. It is therefore necessary to measure the concentration of a diluted solution by using it to titrate a known amount of acid. This is called standardization of the solution. For the titrations, you will need to prepare some 0.1 M NaOH by 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 distilled water. After standardizing this dilute solution, you may 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. The 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. (Note 4)

Calculate the weight of primary standard potassium hydrogen phthalate (KHP = $KHC_8H_4O_4$ MW = 204.2 g/mol) required to give a 35 mL titration using 0.10 M NaOH. Using the analytical balance weigh out three samples of primary standard potassium hydrogen phthalate into 250-mL Erlenmeyer flasks. Add about 35 mL of water to dissolve the solid. Test your buret to make sure 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 0.00 mL, or if you prefer, run the liquid level down below zero and read the initial volume. (Note 6)

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 flask with a stream of distilled water from your wash bottle. The end point is the first faint pink color that persists for 20 – 30 seconds. (Note 7) If the titration was done rapidly wait about a minute before reading the buret to allow time for liquid on the upper walls to drain down. Read the buret to the nearest 0.01 mL.

Titrate the remaining solutions. Calculate the molarity of your NaOH solution, the standard deviation 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 repeat the standardization.

Titration of an Unknown Acid

Test your 25-mL pipet to be sure it runs clean.

Obtain about 150 mL of unknown acid in a 250-mL 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 the relative standard deviation of your replicate results.

REQUIRED MEASUREMENTS

PreLab calculations – with correct answers – must be submitted before you can begin work on the experiment. At the end of the day, you must present YOUR results for the standardization of the NaOH and for the titrations of the unknown acid to your lab TA before leaving. As usual, results include the mean and relative standard deviation (‰) of your replicate determinations of the concentrations of the solutions.

NOTES

1) The water used to make standard 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 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 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 might have been done already.)

4) Check your desiccator to be sure it has good desiccant in the bottom and that the lid seals.

5) Be careful to conserve the 1.0 M sodium hydroxide solution. This solution will also be used in subsequent experiments. If you run out we must prepare more and you will have to repeat the standardization.

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.