INS Chemistry lab for Weeks 7 and 8: Determination of the formula weight of an unknown acid by titration.

Useful Reading: chem. text pp.151-156

Overview: This lab requires the following steps:

1. Working in pairs, groups will prepare an approximately 0.1 M solution of NaOH by dilution of a more concentrated NaOH solution.
2. This exact molarity of the diluted solution of NaOH will be determined by titration against a known mass of a standard acid.
3. The same NaOH solution will be used to titrate a known mass of an unknown organic acid. Since you know the molarity of your NaOH solution and the mass of the unknown acid, you should be able to estimate the formula weight of the unknown acid.

Preparation:

The lab staff will prepare a model set-up at the start of the lab. You will need a burette with a ring stand and clamp, a magnetic stirrer, a magnetic stir bar, and a couple of 100 to 250 mL Erlenmeyer flasks. Be sure that your burette is clean and does not leak by testing it with DI water. You may want to practice controlling the flow from your burette. Set up a station for your own work based on the model set up shown.

We will provide a stock solution of approximately 1 M NaOH. You will need about 250-500 mL of 0.1 M NaOH for this lab. Using a volumetric flask and the technique demonstrated, prepare your dilute NaOH. Be sure that your final solution stays covered, as the solution will absorb carbon dioxide from air over time and lose strength. CAUTION: Remember, you are working with a strong base solution here. Have you labeled your containers?

Rinse your burette with a small amount of your standard NaOH and drain it through the stopcock and the bottom. Repeat this with 2-3 small runs of your NaOH and then fill the burette to near the top. You want your upper level to be in the range of marked volumes on your burette. Use a funnel for these transfers.

Standardization of NaOH with a known acid

The standard acid we are using is potassium hydrogen phthalate (from now on **KHP**). This is a very high purity, monoprotic acid with a formula weight of 204.2 g/mole. We have dried this standard and it will be stored in a dessicator when not in use. Weigh out 3 samples of KHP, each about 0.6-0.8 g. Determine the mass of each sample to the nearest mg or 0.1 mg. Put each sample separately into a clean 250 mL flask, and be sure to known which mass is in which flask.

Add 50-100 mL of DI water to each flask and swirl to dissolve the acid. Add 2-3 drops of phenolphthalein indicator solution to each flask. (For consistency, the same water and indicator volume should be used for all of your titrations.)

Set up the flask under your burette with gentle stirring. Note the starting volume of NaOH solution in your burette to the nearest 0.02 mL, or the best precision possible with your burette. Titrate the acid in the sample by slow addition of your base solution. At the start, the indicator is colorless in acidic solution. You will see the solution turn pink in the areas in which the base is added, but the color will vanish as the acid-base neutralization reaction occurs. As the pink color persists longer and longer, slow the addition of base to single drops. You want to hit the point at which the faint pink color first persists in your flask. It is useful at this point to rinse the sides of your flask with a small amount of DI water to wash any splashed solution on the side of the flask into the reaction. Carefully determine the final volume on your burette, and calculate the volume of solution delivered.

Since you know the mass and the formula weight of KHP used, you can calculate the number of moles of acid in your titration. Since KHP is a monoprotic acid and NaOH provides a single hydroxide per mole, when the solution has been neutralized the same number of moles of acid and base have been added. Now you known the number of moles of base added and the volume in which that number of moles was included. From this data, calculate the molarity of your NaOH dilution.

Repeat this procedure at least 2 more times. Typically, you should expect repeat trials to be within about 1 % of each other. If you have a lot of variation in your trials or you are suspicious of one of your results, you may wish to do addition runs. From your data calculate the mean and the standard deviation for the molarity of your base solution.

Titration of unknown acid.

Now select one of the unknown acids. Be sure to record its identity letter or number. Weigh out samples of 0.15-0.25 g of your unknown, again to the nearest mg or 0.1 mg. (Here, I suggest doing one trial first and observe the volume of base needed. Ideally, you would like to do your titration with 20-30 mL of your base needed. If your first titration is way out of this range, adjust the mass of unknown acid accordingly.)

Transfer the known mass of acid to a clean Erlenmeyer flask and repeat the titration process you used in the standardization runs. Remember to add indicator! (Here, the flask does not need to be dry as you will be adding water. It does need to be free of acid or base and be well rinsed.) Repeat at least twice more to obtain three data sets.

In this case, you now know the strength of the base solution and the volume added, giving you the number of moles of base added. Since at neutrality moles of acid and moles of base added are equal, you know the number of moles of acid originally present. From this data and the mass of unknown acid, determine the weight of acid that provides 1 mole of acidic protons. (This is referred to as the **equivalent weight.** For a monoprotic acid, the equivalent weight and the formula weight are equivalent. What would the relationship between formula weight and equivalent weight be for a diprotic acid?)

From your three trials give the mean equivalent weight for your unknown acid and the standard deviation.

Clean up

Make sure that the burettes have all base removed and are well rinsed after use. The neutralized titration reactions can go down the drain with excess water. The base solution should be neutralized before disposal. We will give you directions for this in the lab.