

Lab 4: Identification of an Unknown Diprotic Acid by Titration

Objective: Look over the experiment and write something appropriate. Remember, this is the scientific purpose of the experiment, not an educational objective.

Prelab: Sketch a theoretical titration curve for phosphoric acid's first two protons (the third doesn't dissociate significantly). Use graph paper at least as fine as 4 squares to the inch. The idea is that this will give you some idea of what to expect for the unknown diprotic acid. Using the amounts and concentrations of acid and base mentioned in the lab, **determine the approximate volumes of base added that correspond to where you will want to shift between collecting data at 0.5 ml and at each drop.** You will also want to calculate the approximate volume corresponding to full neutralization of the diprotic acid, so you will know when to stop titrating. Be warned that the solution concentrations are not very precisely known, so the values you calculate will be approximations.

Materials: Look over the experiment and write something appropriate.

Procedure:

The pH meters have been precalibrated, so we will trust that they won't drift too much in a two hour period.

Place your stirbar in a 100 mL beaker and pipet 10.0 mL of 0.10 M unknown diprotic acid into the beaker. Use a graduated cylinder to measure out 30.0 mL of distilled water and carefully pour the water into the beaker. The purpose of the water is to provide enough depth for the pH electrode to be properly immersed in solution.

Prepare a buret with 0.10 M NaOH (recording the actual concentration), making sure you have enough volume above the 50.0 mL mark for full neutralization plus a little extra because it is approximate. Start the magnetic stirrer, keep the speed down, and measure the initial base volume and the initial pH.

Titrate, recording base volume and pH at appropriate intervals for the different regions of the titration. The first equivalence point should be fairly clear and will allow you to make corrections to your volume approximations, particularly the volume for full titration. Continue collecting data at appropriate intervals until you have added 1.0 mL more than the corrected volume for a full titration.

Rinse and blot the electrode. Empty the contents of the beaker into the waste container, but try to keep the stirbar in the beaker. Rinse both beaker and stirbar thoroughly with tap water and dry them.

Repeat the experiment for a second trial, remembering to check that the buret has enough solution volume above the 50.0 mL mark for a full titration, plus a little more.

Laboratory Hygiene: Once solution is taken from a reagent bottle, it should not be returned to the bottle, so only take what you need. Also, one never pipets directly from the reagent bottle. These measures are to avoid the risk of contaminating the stock solutions.

Waste: All solutions are collected in a waste container for pH adjustment by the lab technician before disposal. Rinse water should not go into the waste container.

Calculations: Each data set must be graphed separately, the finer the divisions, the better. You will be determining values from the graph, so those values will be limited by the precision of the graph paper. On each graph, determine and mark the inflection points. On each graph, mark the equivalent and half-equivalent points and determine and mark the pK_{a1} and pK_{a2} . Since you are taking the base volumes and pK_{a} 's from the graph, you need to show on the graph what those values are. The graphs should be taped into your notebook. Make a table of the data you are getting from the graphs.

For the two titrations:

Determine diprotic acid concentration based on the equivalence points of each titration curve. There should be two on each of the curves, resulting in four concentration values.

Calculate the average and standard deviation for the diprotic acid concentration.

Determine the average and standard deviation for pK_{a1} and pK_{a2} .

Determine which diprotic acid was used. This acid is listed in Table C.1 in Appendix C of your textbook.

Calculate absolute error and percent error for your average pK_{a1} and pK_{a2} .

Conclusion: State the average and standard deviations for the diprotic acid concentration (including its identity), pK_{a1} , and pK_{a2} . For each, make a statement about the magnitude of the standard deviation as compared to the precision of the average.

State the absolute errors for the pK_{a} 's and compare them to the standard deviations. Indicate whether this procedure resulted in pK_{a} 's that were more accurate than precise, more precise than accurate, or of approximately equal accuracy and precision.

State your percent errors for the pK_{a} 's. Percent errors for pK_{a} 's in a titration should be fairly low, approximately 1% is reasonable. How does yours compare?

Comment on the possible causes of any particularly large errors or standard deviations.

Question: Why have you been told "the finer the divisions the better" in this experiment?