Experiment 2B - Buffers

Prelab 2B: Prelab Questions are in the ELN! Don't forget to prepare your procedure!

Part 1 Solution Mixtures – Buffers! (work in pairs)

1. **Acetic Acid/Acetate Buffer**
   Prepare each solution using a graduated cylinder with a lab partner. Measure the pH of each solution. Create a summary table in the results section where you compare the measured values to the calculated values. **Do not discard the buffer solutions. They are needed for part 3.**
   (a) 5.0 mL 0.050 M Acetic Acid + 45.0 mL 0.050 M Sodium Acetate
   (b) 15.0 mL 0.05 M Acetic Acid + 35.0 mL 0.05 M Sodium Acetate
   (c) 25.0 mL 0.05 M Acetic Acid + 25.0 mL 0.05 M Sodium Acetate

2. Measure out 15 mL of 0.050 M Acetic Acid and place in a 50 mL beaker. Fill a buret with 0.1 M NaOH. Using you pH meter, slowly add the NaOH until a pH of 5 is reached. Record the volume of NaOH used. Dilute precisely to 25.00 mL final volume with DI water. Record the new pH.

3. **Carbonate and phosphate buffers**
   Prepare each solution using a graduated cylinder with a lab partner. Measure the pH of each solution. Create a summary table in the results section where you compare the measured values to the calculated values. **Do not discard the buffer solutions. They are needed for part 3.**

   (d) 25.0 mL 0.05 M KH_2PO_4 + 25.0 mL 0.05 M Na_2HPO_4
   (e) 25.0 mL 0.05 M $NaHCO_3$ + 25.0 mL 0.05 M Na_2CO_3

4. **Neutralization of a buffer**
   Divide each of the buffer solutions (solutions a-e) into 25.0 mL portions each in a separate beaker. Each of you will add 1.0 mL of either 0.1 M NaOH or 0.1 M HCl to your buffer solutions, as you did in the previous part of the experiment. Measure the pH that results. Be sure to record all data in both notebooks. Create a summary tables in the results section.

5. **Unknown Acid:**
   Use autotitration to collect data and construct a titration curve of an unknown acid with a known base. Compute the first derivative of the titration curve to locate the equivalence point. From the mass of the unknown acid and moles of titrant, you can calculate the molar mass of the unknown and determine its identity.
   a. Obtain an unknown acid; the exact mass to use will be given in lab.

   b. Using the Hanna autotitrator and standardized 0.1 M NaOH, obtain a titration curve for your unknown acid.

Instructions for using Hanna autotitrator:

1. **Prepare the burette Calibrate the pH meter:**
    a. Remove the storage cap on the working/reference electrode
    b. Loosen up the green thumb screw on the capillary reference electrode
    c. Check that the capillary electrode is filled with electrolyte
    d. Check that the storage cap was still full or half full prior to removing it (rotate the screw up to loosen the screw)
    e. Rinse the working/reference electrode with MilliQ water
    f. Press BURETTE key
    g. Enter # of rinse cycles (suggested: 3), press accept
    h. PRIME BURETTE **(**insert aspiration tube into millQ water and place a waste container under dispensing tip)
2. **Calibrate the pH meter:**
    a. Select "CLEAR OLD CALIBRATION"
    b. Calibrate with 1st buffer
    c. Stir & take 1st calibration measurement
    d. Rinse working/reference electrode
    e. Repeat with additional buffers
    f. Once done with calibration: EXCAPE
3. **Collect data:**
    a. Place tubing into your NaOH solution
    b. PRIME BURETTE Prime the pump & tubing
    c. Visual check: no air bubble in burette
    d. Immerse electrode in your sample solution all the way to the electrode junction
    e. START dosing: Volume delivered, pH and dosing curve will be displayed on screen
    f. BEEP indicates dosage is complete (if it takes more than 25 mL of titrant to reach equivalence, dosage will stop and an error message "level exceeded" will be displayed)
4. **Export data**
    a. Make sure your USB drive is plugged in to the instrument.
    b. From the main screen, press GENERAL OPTIONS
    c. Use the arrow keys to highlight *Save Files to USB Storage Device*
    d. Press SELECT, and the list of files will appear.
    e. Use the left and right arrow keys to select the file type: "report files"
    f. Highlight the name of the report file to be transferred (probably the most recent report file) and press COPY FILE
    g. Transferring a report will automatically transfer the corresponding log file and titration graph.
    h. Press ESCAPE twice to return to the main screen
5. **Cleaning and shut down:**
    a. PURGE BURETTE with the tubing out in air (to empty solutions)
    b. PRIME BURETTE with the tubing in MilliQ water (to rinse the instrument)
    c. PURGE BURETTE with the tubing out in air (to dry to instrument)
    d. Rinse the electrode with MilliQ water
    e. Immerse the electrode in storage solution (slide the storage cap up onto the electrode, then tighten the air-tight screw (screwing down))

13. In excel, plot the titration curve pH vs volume of NaOH. Determine the equivalence point of the titration by also plotting dpH/dV, as discussed in lecture.

14. Determine:
    a. whether the acid monoprotic, diprotic, or triprotic.
    b. the empirical formula for the unknown.
    c. the pKa of the unknown.

15. Based upon this data, do your best to determine the identity of the unknown acid from the list of options provided in class. Write a conclusion in the analysis and results section with supporting evidence as to how you determined your unknown.

Analysis and Results (ELN) Due Feb. 24th:

Add the results from today's lab to experiment 2A. Summarize all results in properly labeled tables and figures with appropriate narrative (1-2 sentences) to explain contents of each summary table or figures. Discussion questions for part 2:

1. Which buffer mixture was most effective at resisting changes in pH? Which buffer mixture was least effective? Why do you think this is?

2. You made one buffer by neutralization, rather than as a mixture of acid and conjugate base solutions. Consider the following questions: Did the volume of NaOH used match what was expected? How much did the pH change when the solution volume was adjusted to the final volume?

3. Determine the identity of your unknown acid based on your titration curve. Discuss the strategies you used to determine the endpoint and explain which you think was most effective.

4. Compare your calculated unknown molar mass, and it's pKas to values found in the literature (or in the textbook). Discuss any possible sources of error.

Don't forget to include a conclusion!