Acid-Base Titrations, Indicators and Buffers

In this experiment you will be given a solution of a base, Na_2CO_3(aq), and a solution of an acid, HCl(aq), but the concentration of only one of these solutions will be known. The goal of this experiment is to determine the concentration of the unknown solution using an appropriate set of data obtained from titrations using three different acid-base (visual) indicator solutions and a pH meter. Visual indicators change color over a relatively narrow pH range, known as the endpoint. When using a visual pH indicator it is important to match the endpoint of the indicator with the expected pH of the equivalence point of the titration being observed. The equivalence point is the point in a titration when the moles of acid and base present in the reaction match the stoichiometry of an appropriately balanced chemical equation.

Safety Concerns:
The solutions of acids and bases used in this experiment are dilute; however, caution is still required. Wear goggles at all times in the lab. Wipe up any spills immediately and rinse the area with water.

Experimental Procedure:

IA. Obtain a burette and make sure that it is clean and does not leak. If necessary, clean the burette with a burette brush and soapy water and rinse it with distilled water. Then rinse it several times with a few milliliters of the stock HCl solution, make sure that the stopcock and burette tip are also thoroughly rinsed with HCl. Fill the burette with the HCl solution making sure that there are no air bubbles and no leaks in the stopcock or tip.

IB. Obtain about 100 mL of stock Na_2CO_3 solution in a clean, dry beaker. Rinse a clean 20 mL pipette with a few milliliters of Na_2CO_3 solution. Then pipette 20.00 mL of the Na_2CO_3 solution into a clean, 125 mL Erlenmeyer flask. Add 4-5 drops of phenolphthalein indicator solution to the Na_2CO_3 solution. Swirl to mix. This solution will be pink and will turn colorless when the endpoint is reached. Place a white piece of paper under the flask so you will be able to easily see when the solution turned colorless.

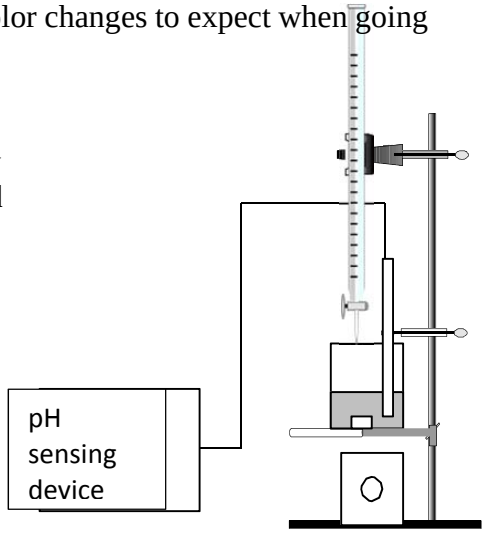
IC. Slowly allow the HCl solution to drain into the Na_2CO_3 solution while swirling the flask. As the endpoint approaches, endpoint color persists longer and longer. Slow down the drain rate as you approach the endpoint which is colorless when phenolphthalein is the indicator. As the endpoint gets closer add HCl drop by drop, swirling the reaction mixture well before the next addition. Stop the addition of HCl as soon as one drop causes the solution to turn colorless and remains colorless. Record the volume of HCl necessary to give the indicator color change that is permanent. If you feel that you overshot the endpoint repeat the experiment. The color changes to expect when going from a basic to an acidic solution are:

Phenolphthalein	pink →→ colorless
Bromothymol blue	blue →→ (green) →→ yellow
Methyl orange	yellow →→ (orange) →→ red

ID. Repeat the titration-using bromothymol blue as the indicator. Record the volume necessary to reach the green color.

IE. Repeat the titration-using methyl-orange as the indicator recording volume to reach the orange color.

Q: Did all three indicators change color when the same volume of acid was added? Are the volumes related? Did all of the indicators have a sharp, easy to detect endpoint?



II.

III. pH Titration: Set up the apparatus as shown in the diagram. In this part of the experiment use a pH meter to monitor the pH variation during titration. Standardize the pH meter using pH 4 and 7 buffers. Review the pH probe video if you forgot how to standardize the pH meter. Use a 150 mL beaker, as there is not adequate room to place the pH probe and the burette tip into of an Erlenmeyer flask. Pipette 20.0 mL of Na_2CO_3(aq) stock solution into the beaker and add ~25 mL of distilled water to the pipetted sample. Also add 4-5 drops of methyl orange indicator to this reaction system. Use a magnetic stirring bar to mix the solution efficiently. The rate of change of the pH with the addition of HCl will vary considerably during the titration. Run a titration by adding ~0.5 mL aliquots of HCl. Allow a few seconds of mixing time before you take readings. Record the pH and exact volume of added HCl during the titration. You will need to make a table in your notebook. Also record the pH and volume of HCl added when the indicator changes its color. As you get close to the equivalence point, start adding the HCl a drop at a time. Continue the titration until the pH reaches a value of 2, which will be well past the endpoint.

Logger Pro has a feature that enables you to determine the equivalence points relatively easily. If you right-click on the vertical axis label you can choose to plot not simply the pH but the first and second derivative of the pH also. At an equivalence point the first derivative is at a maximum and the second derivative goes through zero. You may need to readjust the axis scales to see the data points for the three parameters (pH, 1st deriv.pH, 2nd deriv.pH) at the same time. From the graph, note and record any equivalence points in your laboratory notebook.

Copy the graph using the Snipping Tool accessory and paste it into a Microsoft Word document and then email this file to each member of your group. You will need to include and make reference to the pH titration in your hand-in.

Q: Explain the relationship between the two equivalence point volumes in a chemically reasonable way. Given the observed titration curve, review your data from the indicator titrations and explain your observations. Are all three visual indicators appropriate for this titration? Why or why not? How does your calculation of the unknown concentration change when using each visual indicator? Calculate the unknown concentration using as many endpoints/equivalence points as you feel are valid.

IV. Buffers

Graduated cylinders may be used for measurements in this part for simplicity. When half an equivalent of acid has been added to a sample of carbonate ions, the concentration of the species CO_3^{2-} and HCO_3^- are equal which means that you have produced a good buffer.

IIIA. In a 150 mL beaker, prepare a buffer solution in which the concentration of CO_3^{2-} and HCO_3^- are equal by starting with 50.0 mL of the stock carbonate solution and adding an appropriate volume of HCl(aq) to protonate *half* of the carbonate ions present in solution. This is the concentrated buffer solution. Transfer 25 mL of this concentrated buffer solution to a second beaker and add 40.0 mL of deionized water to make a dilute buffer solution, mix well. You will need two 15 mL samples of each buffer in separate beakers (4 total samples) for testing in the next part of the experiment.

IIIB. Measure the pH of one of the two concentrated buffer solutions. Add 1drop of 1M HCl and record the new pH of the solution. To the second concentrated buffer solution add 1drop of 1M NaOH and record the new pH of the solution.

IIIC. Repeat IIIB for the two dilute buffer solutions.

IIID. Repeat IIIB with two 15mL samples of distilled water with a spatula tip of NaCl added. {This is a blank for reference. The NaCl is necessary to allow conductivity in the water so the pH meter can operate properly but the NaCl does not affect the pH}

Q: ***Were both buffers (concentrated and dilute) equally "good" at limiting the change in pH when strong acid and/or base are added? Explain. Over what range of pH would you expect this buffer system to be "good"? Explain***

Indicator Spectra

Add 50.0 mL of deionized water, 5.0 mL of Na_2CO_3(aq) stock solution, and 10-15 drops of bromothymol blue indicator to a 150 mL beaker and stir gently with a magnetic stir bar. **OPEN UP A SEPARATE COPY OF LOGGERPRO** and set up to record an Absorbance vs. Wavelength spectrum. Fill a cuvette with your carbonate solution and record the Absorbance vs. Wavelength spectrum. ***Return this solution to the beaker***, add 1-2 drops of HCl(aq) to the beaker, let the solution stir for a few seconds, and measure the Absorbance vs. Wavelength spectrum of the solution again by taking some of the solution out and putting it in a cuvette.
Keep all of the spectra on the screen for this entire portion of the experiment so you can see how the spectrum changes. Return the cuvette solution to the beaker and add 1-2 drops of HCl(aq), mix well, and measure the Absorbance vs. Wavelength spectrum. Continue this procedure until you have added 4-6 drops of HCl(aq) ***beyond the endpoint***. Remove the colored background from the spectrum and copy using the Snipping Tool accessory. Paste the spectrum into a Microsoft Word document and email the file to each member of your group.

Q: ***How did the Absorbance vs. Wavelength spectrum change throughout this portion of the experiment?***