Unknown indicators, acids, and buffers

Science, Chemistry

Unknown Indicators, Acids, and Buffers If we knew what it was we were doing, it would not be called research, would it? — Albert Einstein In the following three experiments, you will design your own experiments. We will explain the general problem – the analysis of an unknown – and give you a brief introduction to any necessary theory. Working in a team of students, you will design and carry out every aspect of the experiment. You and your team will have to answer many seemingly mundane questions for yourself. How much solution should you make? What concentration should the solution be? What glassware should you use? You will also have to come up with your own strategy. You will have a total of 5 lab periods to complete 3 experiments: the pKa of an unknown indicator, the identity of an unknown solid acid, and the composition of an unknown acetic acid buffer. We strongly suggest that you do not try to leave early until your group has finished all of the experiments and analyzed all of your data. Do not fall behind, as you will not be given additional time. The suggested timeline is as follows: Week 1: pKa of Unknown Indicator Week 2: Unknown Acid Week 3: Unknown Acid Week 4: Unknown buffer solution. Week 5: Unknown buffer solution I have one very important suggestion: Think before you act! The pre-laboratory questions for these experiments are designed to get you thinking along the right track. Because of time constraints, you must have a clear picture of what you are doing in your experiment before you start. Spending an extra 10 minutes discussing your plans with your group can easily save hours in the lab. Before you take your data, make sure that you know exactly how you will analyze your results. Otherwise, you might forget to make some vital measurements. Also, make sure to record all your observations in your

notebook. Most importantly, have fun. This is what chemistry is all about. – MAH Unknown Acids, Indicators, and Buffers page 2 The pKa of an Unknown Acid-Base Indicator Your group will be assigned a solution containing an unknown amount of an unknown acid-base indicator. In the following, you will design and carry out a series of experiments designed to first qualitatively and then quantitatively measure the pKa of this indicator dye, which is defined to be pK a = âˆ’ log K a . (1) This definition is in direct analogy to pH, which is defined to be pH = âˆ’ log âŽ¡ H + âŽ¤ . âŽ£ âŽ¦ (2) Each group will have access to one pH meter and one visible spectrometer. Because of this, it is important that you work efficiently when making your measurements. After your team decides on an experimental strategy, everyone should be making measurements. For this reason, it is important that you operate as a team in scheduling and sharing equipment. At the end of the experiment, you will share your results with your team members; however, part of your grade will be determined by whether you performed your fair share of the team's Fig. 1: A color wheel summarizes the experiments. relationship between the perceived An indicator dye is just a weak acid (or base) that changes color color of a substance and the color of when it loses (or gains) a proton. For the weak acid case, this light that it absorbs, which is the process can be qualitatively described by the reaction complementary color. The + — approximate wavelengths of the colors (3) HIn H + In Kc = Ka Color 1 Color 2 are also listed. A = Î» A = Î» max 1 max 2 For example, the popular indicator bromthymol blue is yellow at low pH (high [H+]), turning blue-green at pH ~ pKa ~ 7. 0 before becoming dark blue at high pH (low [H+]). This behavior is shown in Fig. 21. 7 in McQuarrie et al.

The visible color of a solution can be quantified by measuring the colors of light that solution absorbs, or its absorbance, using a visible spectrometer. For example, if a solution appears yellow in white light (i. e. normal room light), the solution must be selectively absorbing non-yellow light. This implies that the solution is absorbing the complementary color of yellow, which is violet. On a visible spectrometer, this solution would be expected to have a maximum absorbance in the violet range, which corresponds to wavelengths of ~400-430 nm. Figure 1 summarizes the relationship between the color of light, its Fig. 2: The visible spectrum of bromthymol blue in solutions of varying pH, ranging from highly acidic (yellow) to highly basic (dark blue). The total concentration of bromthymol blue (i. e., [HIn] + [In—]) is the same in each spectrum. Unknown Acids, Indicators, and Buffers page 3 wavelength, and its complement. Looking at Fig. 1, we can surmise that very basic solutions of bromthymol blue, which are dark blue, must be selectively absorb orange light. This corresponds to light with wavelengths in the range of ~600640 nm. Figure 2 shows the visible spectrum of a bromthymol blue in 5 different solutions which range from very acidic (yellow line) to very basic (blue line). The colors of the lines roughly correspond to the perceived color of the solutions. At the very lowest pH, the spectrum displays a single maximum at 431 nm, which corresponds to Î» 1 in Eqn (1). As the pH of the solution increases, the maximum at 431 nm decreases in intensity, which indicates that the concentration of HIn, the undissociated form of bromthymol blue, is decreasing. As this occurs, a new absorption band begins to increase at 615 nm, which corresponds to Î» 2 in Eqn (1). Light at this wavelength is absorbed by In—, the dissociated form of bromthymol

blue. One aspect of Fig. 2 may be confusing: In basic solutions, some spectra show a small secondary maximum near 390 nm that is also attributed to the absorption of In—. Secondary maxima such as this are common in large molecules and can be ignored in this experiment. Chemists use visible spectrometers to quantify color and to relate color to concentration. For example, the most basic solution in Fig. 2 has a maximum absorbance at 617 nm of 2. 678. If this solution were diluted by a factor of two at constant pH, the absorbance at 617 nm (or at any wavelength) would decrease by a factor of two. The new absorbance would thus be 2. 678/2 = 1. 339. Because of this, the absorbance reading (i. e. the quantitative color) can be used to measure relative concentrations of colored solutions using A1 (Î») c1 = , A2 (Î») c2 (4) where A1(Î») is the absorbance of a solution of some species at concentration c1 at some wavelength Î», and A2(Î») is the absorbance of the same species at a concentration c2 at the same wavelength Î». Another example will help to illustrate the use of this equation. Suppose that a 0. 10 M solution of yellow food coloring that has an absorbance of 0. 378 at a wavelength of 445 nm. If you are given a second solution which has a measured absorbance of 0. 0179 also at 445 nm, the concentration of the second solution must be: c2 = = A2 (Î») c1 A1 (Î») (5) 0. 0179 (0. 10M) 0. 378 = 4. 7 Ã—10 —3 M Up to this point, we have only considered light of one specific wavelength even though Fig. 2 shows that both HIn and In— absorb at many different wavelengths. Which wavelengths should we use to measure the concentrations of HIn or In—? Inspection of Fig. 2 reveals one very bad wavelength: 505 nm. Notice that all 5 solutions absorb almost the same amount of light at this wavelength. This is because both HIn and In—

absorb 505 nm light. If we want to quantify the amount of HIn, we should use a wavelength where HIn has a high absorbance, and In— has essentially no absorbance. Conversely, if we want to quantify the amount of In—, we should use a wavelength where In— has a high absorbance, and HIn has essentially no absorbance. One final point deserves mention: the importance of strong absorbance. Figure 2 suggests that any wavelength between 600-700 nm could be used to quantify In—, because HIn does not absorb in this range. Unknown Acids, Indicators, and Buffers page 4 While technically true, this assumption neglects the effects of noise. By measuring the absorbance near the maximum wavelength, the effects of spectrometer noise will be minimized. As a result, the best wavelength for measuring [In—] is approximately 615 nm. Objective Measure the pKa of an unknown acid-base indicator first qualitatively, then quantitatively. Available Equipment and Reagents You may use any equipment in your lab drawer in addition to the following items: Standardized Acid Solution: ~0. 10 M HCl (Exact concentration listed on label.) Standardized Base Solution: ~0. 10 M NaOH (Exact concentration listed on label.) Known Indicator Solution: Bromcresol green (Unknown concentration) Unknown Indicator Solution (Unknown concentration in dropper bottle. Assigned by TA.) 1 pH meter per team 1 visible spectrophotometer per team 50 ml volumetric flasks 50 ml burets 25 ml pipettes Experimental Considerations 1. Dye molecules tend to aggregate (stick together) at high concentrations, complicating their spectrum. For this reason, you should work with dilute dye solutions with maximum absorbances of no more than 0. 2. 2. You will have no way of measuring the absolute concentration of your unknown indicator solution. You will, however,

be able to make up solutions with identical total dye concentrations (i. e., [HIn] + [In—]) by adding the same number of drops of indicator to a constant volume of solution. Experiments 1. Using only your eyes, the stock acid and base solutions, the pH meter, and any additional water that you may require (i. e., no spectrometer!), estimate the pKa of your indicator dye. 2. Design and perform an experiment to find the best wavelengths for quantifying the relative amounts of the dissociated (In—) and undissociated (HIn) states of your unknown indicator dye. You will use the same spectrometers that you used earlier in the course. Before trying your procedure of your unknown indicator, you must test your procedure on bromcresol green. 3. Design and perform an experiment to accurately measure the pKa of a your unknown indicator using the visible spectrometer. At each pH that you investigate, you should measure the absorbance of your solution at two wavelengths. Test this procedure on the bromcresol green solution, which has a pKa of 3. 46. 4. Determine the pKa of your unknown indicator using the procedure developed above. Safety Considerations If the solutions come in contact with your skin, rinse the affected areas under running water. Waste Disposal All chemical waste generated during this experiment should be flushed down the sink. Unknown Acids, Indicators, and Buffers page 5 Laboratory Report All lab reports must be written individually. Data that are recorded as a group should be attributed in the lab report to all members of the group. Data that are recorded by a specific group member and shared with the group must be explicitly attributed to the group member who did the work. All text, tables, and figures in your lab report should be prepared by you; copying these materials from another student or sharing your materials with another

student is a violation of the Cornell Code of Academic Integrity. Your report should have the usual cover sheet and should contain an abstract, an introduction, an experimental section, and a results and discussion section. In the abstract, you should mention the experimentally observed pKa for your dye as well as the wavelengths that you used to quantify the dissociated and undissociated forms of the dye. Your experimental section must contain a complete description of your experimental procedure. Any Chem 2080 student should be able to reproduce your experiment after reading your lab report. This section does not have to be long, but it does have to be complete. The results and discussion section should consist of two parts. First, you should prove that your experimental procedure works by presenting your analysis of bromcresol green. Following this, you should present your analysis of your unknown indicator. Discuss how and why you selected your analysis wavelengths. Please include a qualitative discussion of the possible sources of error in your experiment. Unknown Acids, Indicators, and Buffers page 6 The Identification of an Unknown Solid Acid Your group will be assigned an unknown, monoprotic solid acid drawn from the list in Table 1. Your goal is to design and perform two titration experiments that will allow you to identify the acid from a list of known acids. You should base your identification on a measurement of the molar mass and the pKa of your unknown. Since you will be designing your own experiment, it is imperative that you test your procedures on a known solid acid. A supply of potassium hydrogen phthalate (KHP) will be made available for this purpose. KHP has a molar mass of 204. 23 g/mol and a pKa of 5. 408. Your first titration experiment should be designed to measure the molar

mass of your unknown acid, whereas the second experiment should be designed to measure the pKa of your acid. One of your experiments must be done with one of the known indicator dyes (i. e., no pH meter). The other experiment must be done with a pH meter (i. e., no indicator dye). Since your experiments will be quantitative, you need to make sure that you use a quantified amount of acid. You will therefore have to decide how you are going to perform this quantification. Will you weigh out a precise amount of unknown acid for each experiment or will you make up a stock solution for many experiments? Remember that if you decide to make up a stock solution, you will need to use a volumetric flask. To transfer a precise amount of solution, you must use a pipette. (Graduated cylinders are only used for approximate measurements. Precise volume measurements can only be made with volumetric flasks, pipettes, or burets.) The first question to ask is what experiments are you and your team going to perform? Once you decide on your experiments, split your team into two sub-teams and test the experiments on KHP. Make sure that your experiments give the correct answer for KHP before you start working with your unknown. The second question to consider is how concentrated do you want your solutions to be? The choice is entirely up to you, but there are some factors to consider: - The stock HCl and NaOH solutions are approximately 0. 10 M. Exact concentrations will be posted in the lab. - Your buret holds 50. 0 ml of solution and is labeled in 0. 1 ml increments. You should make measurements to the nearest ~0. 01 ml by interpolating by eye (see Fig. 12. 14 in McQuarrie et al.). For accuracy, you should try to perform titrations that require a total of ~10—20 ml of added acid/base. A titration that requires 0.

9 ml will be difficult to measure quantitatively, whereas a titration that requires 75 ml will be both wasteful and less accurate. Since you know the molar mass of KHP, calculate the number of grams of KHP that you will need to make up the solution that you want. Since you don’t know the molar mass of your unknown acid, your best bet is to use the same mass of unknown. When you make your solutions, do not try to add exactly the amount of acid you calculated. Instead, weigh out approximately the correct amount of Name Benzoic acid Chloroacetic acid p-Chlorobenzoic acid Diphenylacetic acid 2-Furoic acid Glycolic acid Hydrocinnamic acid Iodoacetic acid l-Lactic acid dl-Mandelic acid Potassium bitartrate Potassium hydrogen phthalate Potassium phosphate monobasic Suberic acid Sulfanilic acid o-Toluic acid Trimethylacetic acid Formula C 7 H6 O2 C 2 H3 ClO 2 C 7 H5 ClO 2 C 14H12O2 C 5 H4 O3 C 2 H4 O3 C 9 H10O 2 C 2 H3 IO 2 C 3 H6 O3 C 8 H8 O3 C 4 H5 KO6 C 8 H5 KO4 H2 KPO 4 C 8 H14O 4 C 6 H7 NO3 S C 8 H8 O2 C 5 H10O 2 Molar Mass (g/mol) 122. 12 94. 50 156. 57 212. 25 112. 08 76. 05 150. 18 185. 95 90. 08 152. 15 188. 18 204. 23 136. 08 174. 20 173. 19 136. 15 102. 13 pK a 4. 204 2. 867 3. 986 3. 939 3. 164 3. 831 4. 664 3. 175 3. 858 3. 37 4. 36 5. 408 7. 2 4. 512 3. 227 3. 9 5. 031 Table 1: Properties of some solid acids. Unknown Acids, Indicators, and Buffers page 7 acid, then record the exact amount in your notebook. For example, if you are aiming for 0. 5 g of KHP, an actual value 0. 4893 g or 0. 5217 g is fine. Also, some solid acids are more soluble than others. You may need to heat your solution gently to get the desired amount of acid into solution. It is imperative that all of the acid dissolves. Otherwise, your experimental results will be flawed. When you perform a titration, do not try to fill your buret exactly to 0. 00.

Instead, record the starting volume (e. g., 1. 75 ml) and the final volume (e. g., 27. 85 ml), finding the total volume by difference (here, 26. 10 ml). Objective Identify an unknown solid acid from two experiments, one using an acid-base indicator and one using a pH meter. Available Equipment and Reagents You may use any equipment in your lab drawer in addition to the following items: Standardized acid solution: ~0. 10 M HCl (Exact concentration listed on label.) Standardized base solution: ~0. 10 M NaOH (Exact concentration listed on label.) Indicator solutions: Methyl orange, thymol blue, and phenolphthalein (Unknown concentrations. See Fig. 21. 7 in McQuarrie et al. for properties.) Unknown solid acid (Pure solid. Assigned by TA.) 1 pH meter per team 1 visible spectrophotometer per team 100 ml volumetric flasks 50 ml burets 25 ml pipettes Safety Considerations If the acids or bases come in contact with your skin, rinse the affected areas under running water. Some may wish to wear gloves when handling the solid acids, as some are caustic. Experimental Considerations 1. Read Section 12-6 in McQuarrie et al. before you design your experimental protocol or come to lab. 2. One of your titration experiments, either to determine molar mass or pKa, must use the indicator dye, but not a pH meter. The other experiment must use a pH meter, but not an indicator dye. 3. Each titration must be performed at least 3 times for accuracy. This experiment will require at least 12 titrations: 3 for the molar mass determination of KHP, 3 for the pKa determination of KHP, 3 for the molar mass determination of the unknown acid, and 3 for the pKa determination of the unknown acid. Experiments Note: Everyone should individually perform at least one experiment to determine molar mass and one experiment to determine pKa. Unless there

are unforeseen problems, everyone in the team should measure both the pKa and the molar mass of the unknown acid (but not necessarily of the known acid). For accuracy, your team should make multiple measurements of both the pKa and the molar mass of your unknown. If all of the experiments are in good agreement, you will be able to average your results to reduce the effect of experimental error. If some of the experiments give anomalous results, you will need to consider your strategy carefully. Your group will only have one pH meter, so you should try to get started on experiments involving the pH meter as soon as possible. 1. On the first day, your group should decide on an experimental strategy. (Before coming to lab, you should have already developed a plan yourself. The discussion among your teammates should be Unknown Acids, Indicators, and Buffers page 8 brief, as time is limited.) In designing your experiments, remember that one experiment must use a pH meter (no indicator dye), while the other must use a known indicator dye (no pH meter!) 2. Once you decide on a strategy, split your team into two groups and try your proposed experiments on KHP. Compare the results of these experiments to the known molar mass of KHP. If you cannot reproduce the known results, rethink your strategy. From these measurements, you will also get a rough idea of your experimental accuracy, which will be important in determining the identity of your unknown. 3. Once you have completed the KHP experiments, you will need to determine the molar mass and pKa of your unknown acid. Ideally, everyone in the group should make at least one determination of pKa and one determination of molar mass. Safety Considerations If the solutions come in contact with your skin, rinse the affected areas under running water.

Waste Disposal All chemical waste generated during this experiment is to be flushed down the sink. Laboratory Report All lab reports must be written individually. Data that are recorded as a group should be attributed in the lab report to all members of the group. Data that are recorded by a specific group member and shared with the group must be explicitly attributed to the group member who did the work. All text, tables, and figures in your lab report should be prepared by you; copying these materials from another student or sharing your materials with another student is a violation of the Cornell Code of Academic Integrity. Your report should have a cover sheet and should contain an abstract, an introduction, an experimental section, and a results and discussion section. In the abstract, you should mention the identity of your unknown and your percentage error in determining the pKa and the molar mass. Your experimental section must contain a complete description of your experimental procedure. Any Chem 2080 student should be able to reproduce your experiment after reading your lab report. This section does not have to be long, but it does have to be complete. The results and discussion section should consist of two parts. First, you should prove that your experimental procedure works by presenting your analysis of KHP. Following this, you should present the analysis of your unknown acid. Both analyses should include estimates of your experimental error (assuming that you correctly identified your unknown acid). Remember that the percentage error is defined to be % error = Measured value — True value Ã — 100% . True value (1) Please include a qualitative discussion of the possible sources of error in your experiment. Unknown Acids, Indicators, and Buffers page 9 The Composition of an Unknown Buffer Your group will be

given a sample of a buffer containing only unknown quantities of acetic acid (HAc), sodium acetate (NaAc), and water. Acetic acid dissociates in water, HAc H + +Ac — pKa = 4. 74 where Ac— is the common abbreviation for the acetate ion, CH3COO—. Your goal is to design and perform an experiment to determine the molar concentration of HAc and NaAc in this solution. You may use any equipment that you like; however, your team will again have only one pH meter. For this reason, you should carefully consider whether or not you actually want to perform a titration with the pH meter. There are a number of different ways of solving this problem, some requiring pH meters and others not. You should carefully consider both the ease and the accuracy of your chosen method. Objective Measure the concentrations of acetic acid and sodium acetate in an unknown buffer solution. Available Equipment and Reagents You may use any equipment in your lab drawer in addition to the following items: Standardized Acid Solution: ~0. 10 M HCl (Exact concentration listed on label.) Standardized Base Solution: ~0. 10 M NaOH (Exact concentration listed on label.) Indicator Solutions: Methyl orange, thymol blue, and phenolphthalein (Unknown concentrations. See Fig. 21. 7 in McQuarrie et al. for properties.) Known Buffer Solution: ~0. 05 M HAc/NaAc solution (Exact concentration listed on label) Unknown Buffer Solution (Assigned by TA.) 1 pH meter per team 1 visible spectrophotometer per team 100 ml volumetric flasks 50 ml burets 25 ml pipettes Experiments You should design and perform experiments to measure the concentrations of both components of your unknown buffer. Before performing any experiments on your unknown, you must test your procedure on the known buffer solution. Safety Considerations If the solutions come in contact with

your skin, rinse the affected areas under running water. Waste Disposal All chemical waste generated during this experiment is to be washed down the sink with plenty of water. Unknown Acids, Indicators, and Buffers page 10 Laboratory Report All lab reports must be written individually. Data that are recorded as a group should be attributed in the lab report to all members of the group. Data that are recorded by a specific group member and shared with the group must be explicitly attributed to the group member who did the work. All text, tables, and figures in your lab report should be prepared by you; copying these materials from another student or sharing your materials with another student is a violation of the Cornell Code of Academic Integrity. Your report should have a cover sheet and should contain an abstract, an introduction, an experimental section, and a results and discussion section. Your experimental section must contain a complete description of your experimental procedure. Any Chem 2080 student should be able to reproduce your experiment after reading your lab report. This section does not have to be long, but it must be complete. The results and discussion section should consist of two parts. First, you should prove that your experimental procedure works by presenting your analysis of the known buffer solution. Following this, you should present your analysis of your unknown buffer solution. In this section, include a discussion of possible experimental errors. If possible, suggest improvements to your experimental procedure.