QUALITY CONTROL AT KALITY KRUNCHERS DILL PICKLE FACTORY

INTRODUCTION

Description

This is a two week experiment. During the first lab period, students determine the conditions for a precise, reproducible titration of a brine solution with sodium hydroxide and the indicator, phenolphthalein. During the second week, students titrate pickle juice. They need to solve the problem of the interfering color of the pickles and decide if a more dilute solution of sodium hydroxide would be appropriate.

Goals for This Experiment

The goals for this experiment are to have students:

1. practice the skill of titrating,
2. experience the skills and patience that are needed to obtain less than 1% error in measurements,
3. design a procedure in response to a question posed by the scenario,
4. work with reporting data in the proper number of significant figures, and
5. evaluate a procedure that was developed by another student.

Recommended Placement in the Curriculum

This pickle juice titration laboratory would be best implemented towards the middle or end of a first semester General Chemistry laboratory. The students should have previous experience with titrations, but do not need to be experts at titrating. In addition, students will appreciate the lab more if they have designed a procedure prior to this lab experiment.

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SCENARIO

Kality Krunchers Dill Pickles are produced and advertised to be crunchy, not soft or rubbery. These pickles are "quick" pickled with a hot brine of vinegar, water, salt and spices. Alum and calcium chloride are also added to produce a firm pickle. The vinegar and salt draw some of the water out of the cucumber producing a "pickle." The vinegar's acidity prohibits bacterial growth and provides shelf life.

Lately, some Kality Krunchers Dill Pickles have been reported to be unpleasantly soft. A manager wonders if this is due to uneven ripeness of the cucumbers and wonders if checking the degree of acidity of the pickling brine during manufacture might give pickles uniform superiority. Pickle packing is seasonal work, and the manager has asked you, a summer employee/college student if you think this is feasible. The manager even offers "technical assistance." His daughter, a high school senior who has just completed high school chemistry, is willing to work on the problem as well.

BACKGROUND

Deja vu! Been there, done that! You recall the titration of acetic acid and sodium hydroxide solution from your chemistry class, but what were the concentrations of the acetic acid and sodium hydroxide solutions? You are pretty sure that vinegar is not a very concentrated acetic acid. A call to your former chemistry professor indicates that you should probably try to find some "official" method to start with. The professor suggests the method published by AOAC, the Association of Official Analytical Chemists. A look in their book, *Official Methods of Analysis*, reveals a titration method for acidity of vinegar, fruit products, catsup, etc.

YOUR TASKS

- Your first task is to determine <u>the conditions for a precise, reproducible titration</u> of the starting brine, (50% water, 50% vinegar and salt) with a sodium hydroxide and phenolphthalein indicator. Does 0.5 M sodium hydroxide or 0.1 M sodium hydroxide work better? (AOAC suggested 0.5 M for vinegar but 0.1 M for fruit products.)

- Next, the problem of the interfering color of the final pickle product needs to be solved. (People expect pickles to be green.) Is it possible, in spite of the green color, to titrate the green dill pickle brine with phenolphthalein reproducibly using the conditions from the initial brine titration method? Or would it be better to dilute with water a sample of the dill pickle brine taken after the pickling process is over? If the sample for titration is diluted, should a more dilute titrant, such as 0.05 M sodium hydroxide then be used? (AOAC suggested this approach for colored cider type vinegars.)

- Because you want to develop a method the manager's daughter can perform, you need to prepare a procedure that will be given to another worker in the laboratory to use and see if the same results are obtained. You will give your written procedure to your instructor, who will have another person evaluate the method. You will also be asked to evaluate the procedure of another student.

SUGGESTIONS FOR PROCEDURE

1. You will have available the following solutions:
   standardized solutions of both 0.5 M aqueous NaOH and 0.1 M aqueous NaOH
   model pickling solution (for task # 1)
   phenolphthalein indicator
   pickle juice from commercial dill pickles

2. You will have a maximum of 100.0 mL of pickle juice to use for your experiment. Make sure you use it wisely. **Before you begin the work,** make very sure that you understand the titration you have planned (identify amounts and concentrations of base).

3. Sample sizes for the brine solution and pickle juice should be between 10–20 mL. Ideally, the amount of titrant should be about 20 mL/sample.

4. Perform at least four titrations in task 1 so that you can show an experimental error no greater than 1% of the mean concentration.

5. **Reference materials:** Directions for titrating brine solution from the AOAC, information on the process of pickling, and a recipe for pickling solution from *Joy of Cooking* are posted on the Chem 144 bulletin board just outside the laboratory. A description of the technique and methods of calculations used with acid/base titrations is given on pages 172–173 of your text. You might find the information useful.

SAFETY NOTE: The 0.5 M NaOH solution is sufficiently concentrated to be harmful to your eyes and skin. Be very careful pouring it, making sure that you keep the process of filling the buret **below** eye level. If any solution should get into your eyes or onto your skin, wash the area with large amounts of water and inform your instructor.

You will have two weeks to complete these tasks. As you complete each task, show your work and your plans for the next step to your instructor.

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INSTRUCTOR NOTES

Time Required

The laboratory experiment takes two concurrent lab periods to complete. In our experience, most students used the full lab period each time to complete their assigned tasks. In the first lab, students learn to titrate within 1% error using model pickling juice (a clear and colorless brine solution that contains 1:1 white vinegar and water plus salt as noted in the materials list). In the second lab, students titrate the pickle juice obtained from a deli or store-bought pickles. The challenge in the second week is the color of the solution and the problems it presents in detecting the change in color at the endpoint. (In this experiment, phenolphthalein is used as the indicator and the color change in the model pickle juice is from colorless to light pink.)

Group Size

Students work individually to give them the opportunity to improve their titration techniques. You could have students bring in their own pickle juice sample and compare results.

Materials Needed

First lab

per class (20 students):

- 3 L of model pickling juice (brine solution): add 1.5 L of white vinegar to 1.5 L of water (can be tap water). Add 12 cups of kosher salt. Stir to dissolve.
- 3 L of 0.5 M aqueous NaOH that has been standardized (molarity known to four significant figures): To make the solution, add 60 g of NaOH pellets to 2.5 L of H_2O. Once the sodium hydroxide has dissolved, dilute the solution to 3 L with H_2O. To standardize the solution, used potassium hydrogen phthalate (KHP). First, dry the KHP in an oven for 1 hour at 120°C. Next, accurately weigh out approximately 0.5 g of KHP (to four decimal places if possible) and place it into a 250-mL Erlenmeyer flask. Add 50 mL of distilled water and swirl the contents until the KHP is completely dissolved. Titrate the solution with sodium hydroxide and calculate its concentration. Repeat the procedure at least two more times to ensure an accurate and precise concentration.
- 3 L of 0.1 M aqueous NaOH that has been standardized (molarity known to four significant figures): To make the solution, add 12 g of NaOH pellets to 2.5 L of H_2O. Once the sodium hydroxide has dissolved, dilute the solution to 3 L with H_2O. Standardize the solution as above.
- 100 mL of phenolphthalein indicator: Add 0.1 g of phenolphthalein solid to 100 mL of 95% (or better) ethanol.

per student:

- buret
- 10-mL pipet or a second buret (to dispense the pickle juice/brine solution)
- two 250-mL Erlenmeyer flasks

Second lab

per class (20 students):

- 3 L of 0.1 M aqueous NaOH that has been standardized (molarity known to four significant figures) using the above method.
- 3 L of 0.05 M aqueous NaOH that has been standardized (molarity know to four significant figures) using the above method. To make the solution, add 6 g of NaOH pellets to 2.5 L of H_2O. Once the sodium hydroxide has dissolved, dilute the solution to 3 L with H_2O.
- 100 mL of phenolphthalein indicator: Add 1 g of phenolphthalein solid to 99 mL of 95% (or better) ethanol.
- 3 L of commercial pickle juice

per student:

- buret
- 10-mL pipet or a second buret (to dispense the pickle juice/brine solution)
- two 250-mL Erlenmeyer flasks

Notes:

✔ For best results, use either kosher salt or chemistry lab salt in the brine solution rather than table salt. Table salt has an anti-caking agent (silica) that will not completely dissolve and, as a result, clouds the solution.

✔ You may be able to obtain pickle juice from a local deli or sandwich shop that serves pickles.

✔ As a part of the inquiry aspect of this lab, students are asked to decide which concentration of NaOH they want to use. The first week both 0.5 M and 0.1 M NaOH solutions are made available to the students. Students may pick one concentration and then decide to switch to the other. Whichever concentration they choose, they need to make sure that they are fulfilling all the criteria. (Sample solution amount is 10–20 mL and amount of titrant used is between 10–50 mL.) Likewise, the second week both 0.1 M and 0.05 M NaOH solutions are available. You may wish to have 0.5 M NaOH available also. Students will probably choose to start with the concentration of NaOH that they used the first week. However, the pickle juice has a lower acid concentration than the brine solution. Thus, if students choose to use the 0.5 M NaOH, they will probably use less than 10 mL of titrant to reach the endpoint. Remember, no matter what concentration students choose, let students come to the realization that the concentration is or is not appropriate based on the requirements of the procedure.

Safety, Disposal, and Special Handling
Review the Material Safety Data Sheet (MSDS) of any chemical used in the experiment for information regarding safety and handling. Dispose of waste according to your local ordinances.

Points to Cover in Pre-Lab *(The First Week)*

- The students' goal in Part I is to learn to titrate precisely. Their frustration can be avoided if they know that the main purpose of Part I is to give them the opportunity to improve their titration technique.
- Review what it means to titrate a sample.
- Remind students that burets can read to the nearest ±0.01 mL, and pipets measure to four significant figures (if the volume ≥10.00 mL).
- Point out to students that phenolphthalein is colorless in an acidic solution and pink in a basic solution.
- Tell students that they can perform the first titration rather quickly to get an idea of how much titrant is need to reach the endpoint. The remaining titrations are done slowly to ensure accuracy and precision.
- You may need to define a monoprotic acid (an acid that donates a single proton, such as HCl).
- Remind students that good lab procedures include recording **every** sample they do, even the samples that are titrated incorrectly. Show them that good lab procedure involves drawing one line through the discarded data and indicating the reason for discarding the data.
- Instruct students on the proper way to fill and rinse burets when going from a more concentrated to less concentrated solution (i.e., from 0.5 M NaOH to 0.1 M NaOH).
- Point out to students that they have to be comfortable with the titration and the color change when titrating the ideal solution (the brine solution) since the following week the color change will not be as easy to detect (pickle juice is colored, the brine solution is colorless.)
- Discuss with students the conditions necessary to obtain an accurate and precise titration. For example, how many significant figures are required and what conditions must be met to get the proper number of significant figures (i.e., the sample solution volume should be at least 10.00 mL, the amount of titrant used should be at least 10.00 mL, etc.)?
- Show students the pickle juice and ask them what the challenge will be when titrating it (detecting the color change of the endpoint).

Points to Cover in Pre-Lab *(The Second Week)*

- Ask students what they learned from the experiment in the first week.
- Ask students how the pickle juice will differ from the brine solution and how they plan to address the differences.
- Ask students what they might tend to do when titrating the pickle juice (overtitrate).
- Remind students of the parameters for titrating (sample volume needs to be at least 10.00 mL, proper number of significant figures, etc.)

Likely Play-Out of Lab *(The First Week)*
Students will most likely want to use the 0.5 M NaOH to titrate the brine solution. After titrating one sample, some students will decide to switch to the 0.1 M NaOH. Let students discover and decide for themselves which solution to use. Just make sure that they are staying within all the parameters that were discussed in the pre-lab. Most of our students performed 6–8 titrations. Errors ranged from about 40% all the way down to 0.6%. Only careful students achieved less than 1% error; the remaining students became frustrated. Remind frustrated students that this week in lab provides them the opportunity to practice and improve their technique. Many of these students greatly improved their percent error the following week and even achieved less than 1% error.

Likely Play-Out of Lab *(The Second Week)*
Students need to have their plan of action written down and turned in before the beginning of the second week of lab. We had students turn in their write-up at the beginning of pre-lab. You may decide to have them turn it in a couple of days before. Whatever you decide, you need to review their plan and make sure it appears reasonable to get the job done and is described in sufficient detail to show that the student understands the task.

Most students will want to use whatever concentration of sodium hydroxide they used the first week. In the background information provided, it states that pickles are "placed in brine for a short time to draw out moisture that would dilute the vinegar." This provides them the clue that the pickle juice will be lower in concentration than the brine solution. Therefore, a lower concentration of sodium hydroxide is needed to titrate the sample within the given parameters. Again, let the students discover the concentration of NaOH that yields the best results within the parameters required. We found that both the 0.1 M and 0.05 M NaOH worked well. (Approximately 15–25 mL of 0.1 M NaOH or 30–50 mL of 0.05 M NaOH were needed to titrate the pickle juice depending on the sample size.) One student decided to dilute the pickle juice hoping that the interfering color would also be diluted and thus detection of the endpoint would be easier. He could detect the endpoint more easily, but found a different concentration for each method (see below).

Once students have gone through their procedure, refined it, and shown that it is reproducible, they need to write the procedure on a piece of paper and give it to another student to evaluate. Students are not allowed to ask for clarification from each other. This process tends to be an eye opener for a lot of the students, especially if they get a procedure from a student that is vague. Several students in our lab evaluated poor procedures and came to the instructor for help. They were told to do what they could, and if they couldn't finish the experiment because of the lack of instructions, to address this in the evaluation of the procedure.

Instructor Suggestions

- Some students were very creative when designing their procedure for the titration of the pickle juice. One student wanted to put the indicator in the base and titrate with the pickle juice until the pink color disappeared. Encourage them to try their ideas, even if they seem unconventional; students will still learn from the experiment. He performed the titration his way and the conventional way and discovered that the calculated concentrations for each method did not agree (0.1317 M for the "reverse" titration vs. 0.1267 M for the "traditional" titration). This led to a meaningful discussion of what could be the cause for the different calculated concentrations in the two methods. It was exciting for the instructor and rewarding for the student.
- You may wish to make some or all of the following information available to students:
  - ➢ a pickle recipe from any common cookbook;
  - ➢ the pickle section from *On Food and Cooking: The Science and Lore of the Kitchen,* by Harold McGee (MacMillan Publishing Company, 1984) or a similar "kitchen science" publication; and
  - ➢ directions for titrating brine solution from the AOAC.