NSTA 2025
Philadelphia, PA

Green Chemistry in Action: Conductometric Titration with Household Chemicals

Experiments

Eco-Friendly Precision: Standardizing Vinegar through a Green Chemistry Titration

- Go Direct® pH Sensor
- Go Direct Drop Counter

Green Chemistry in Action: Conductometric Titration of Ammonia with Vinegar

- Go Direct Conductivity Probe
- Go Direct Drop Counter

Workshop Presenter

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Eco-Friendly Precision:

Standardizing Vinegar through a Green Chemistry Titration

Acetic acid (CH_3COOH), also known as ethanoic acid, is perhaps the most important of the carboxylic acids. It is used as a reagent in the manufacture of a wide range of products including textiles, plastics, photography, and medicine. In the home, acetic acid is valued as an ingredient in food preparation as well as a multi-task cleaning aid. A dilute, aqueous solution of acetic acid goes by another name: vinegar.

The concentration of acetic acid in vinegar can vary depending on the type, brand, and intended use. In grocery stores, a product labeled “pickling vinegar” will contain a higher concentration of acetic acid, required for its use in pickling cucumbers and other vegetables.

The purpose of this experiment is to determine the precise concentration of acetic acid in a specific brand of commercial vinegar. You will do this using a laboratory technique called titration, which involves the observation and measurement of a carefully controlled chemical reaction. In this case, the base will be another commercially available substance called slaked lime [calcium hydroxide, $Ca(OH)_2$]. You will use a pH sensor and a tool called a drop counter to observe and measure the real-time change in pH that occurs as $Ca(OH)_2$ is added incrementally to a specific volume of CH_3COOH.

Aside from being available at grocery stores and hardware/building supply stores, both vinegar and slaked lime are relatively safe and environmentally friendly chemicals. As such, this experiment not only provides a practical application of titration techniques but also highlights the importance of green chemistry by using less hazardous reagents.

Pre-Lab Analysis

1. **Chemical Reaction and Calculations**
    - Write a balanced chemical equation for the reaction of acetic acid (in vinegar) with calcium hydroxide (slaked lime).
    - In most store-bought vinegar, the concentration of acetic acid is 5% by volume. Calculate the theoretical molarity of acetic acid in store-bought vinegar.
    - Assuming the theoretical molarity of acetic acid in store-bought vinegar, calculate the mass of calcium hydroxide needed to completely react with 10 mL of it.
    - How will you ensure your calculations are accurate?
    - How do you expect these theoretical values to compare with actual values you will measure? How will you compare them?
2. **Observation and Measurement**
    - You will be using a pH sensor and live data collection to monitor your titration reaction. Think about the important observations you will need to make to confirm the reaction is complete.

- What changes will you look for in the reaction mixture?
- What changes will you look for in the data? Sketch a drawing of the expected results. Make sure to label your graph completely.
- How will you determine the endpoint of the reaction?

3. **Green Chemistry Application** (Note: If you are unfamiliar with green chemistry, search the internet for “ACS green chemistry” to find a good resource.)
    - Consider how using vinegar and slaked lime aligns with the principles of green chemistry. Why are these reagents considered less hazardous?
    - Discuss how you can minimize waste and environmental impact during the experiment including the use of data collection technology and ensuring that all the materials used are biodegradable and safe for the environment.

Materials

- computer, Chromebook™, or mobile device
- Vernier Graphical Analysis® app
- Go Direct® pH Sensor (GDX-PH)
- Go Direct Drop Counter (GDX-DC)
- Stir Station and magnetic stirring bar
- balance, 0.001 g
- test tube clamp
- (2) 250 mL beakers
- 10 mL graduated cylinder
- commercial vinegar solution of unknown concentration
- commercial slaked lime (calcium hydroxide)
- wash bottle with distilled water

Investigation

Set up for data collection

1. Launch Graphical Analysis and connect your Go Direct pH Sensor and Go Direct Drop Counter.

2. Prepare the Drop Counter for data collection.
    a. Mount the Drop Counter to the Stir Station as shown in the illustration.
    b. Click or tap the Volume meter and choose Calibrate.
        - If the calibration value (drops/mL) is unknown, follow the onscreen instructions under Automatic to calibrate your Drop Counter. You will need a 10 mL graduated cylinder.
        - If the calibration value (drops/mL) is known, click or tap **Manual** and enter the value.
    c. Click or tap **Apply**.

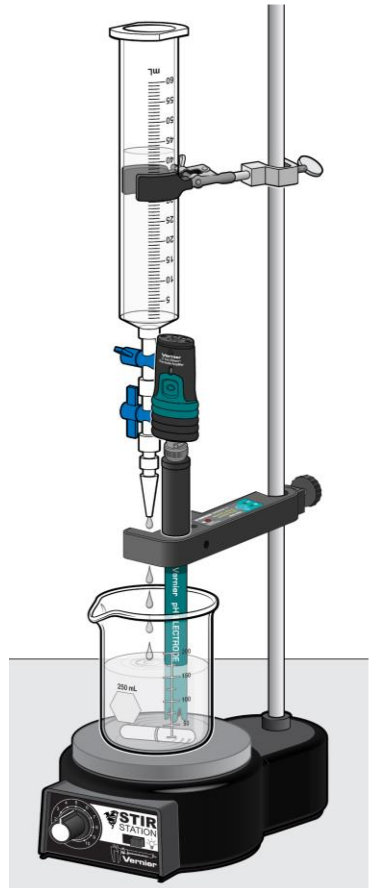

Figure 1

Prepare for the titration

3. Fill the Drop Counter reservoir with vinegar solution of unknown concentration.

4. Align the Drop Counter reservoir with the opening of the drop counter. Test the drops over a 250 mL beaker centered on the Stir Station.

5. Adjust the drop rate of the Drop Counter to about 1 drop every 2 seconds. Use the top valve to adjust the drop rate and the bottom valve to stop and start the drip.

6. Remove the 250 mL beaker. Then, place a clean 250 mL beaker on the Stir Station and add the magnet to the beaker.

7. Place the pH sensor in the Drop Counter. Adjust the beaker as needed to make sure the magnet doesn't strike the sensor.

8. Weight out and record a mass of calcium hydroxide very close to what you determined in the Pre-Lab Analysis and place it in the 250 mL beaker on the Stir Station. **Note**: The mass does not need to be exactly the same as that from the pre-lab, but you do need to know the exact mass you weighed out for the later calculations.

9. Add about 50 mL distilled water to the beaker on the Stir Station.

10. Set the Stir Station to a speed that is stirring briskly (medium to high setting), but that doesn't create splashing.

Collect titration data

11. Collect data.
    a. Start data collection. No data will be collected until the first drop goes through the Drop Counter slot.
    b. Fully open the bottom valve—the top valve should still be adjusted so drops are released at a rate of about 1 drop every 2 seconds.
    c. When the first drop passes through the Drop Counter slot, check the graph or table to see that the first data pair was recorded.
    d. Stop data collection when an acidic pH value stops changing.
12. Dispose of the beaker contents and reset for a second trial, if necessary. Start data collection when you are ready. **Note**: The first data set is automatically stored.

Post-Lab Analysis

1. How many trials did you conduct? What was the mass of calcium hydroxide used for each trial?
2. From the graph for each trial, determine the precise volume of vinegar that was used. Explain how you obtained this value. Include screenshots of your data.
3. Using the balanced equation for this reaction, calculate the moles of acetic acid needed to completely neutralize the mass of calcium hydroxide used in each trial.
4. Calculate the molar concentration of vinegar for each trial.
5. In your pre-lab analysis, you calculated the theoretical concentration of acetic acid in store-bought vinegar (5% by volume). Compare this value to the concentration you calculated from your experiment. Determine the percent error between the theoretical and experimental values.
6. Discuss the percent error value you obtained for this experiment. Consider the following: Is the error higher or lower than what you would expect from traditional titrations with a strong acid and strong base? What are some possible sources of error? How can you verify the concentrations you determined are correct?
7. You will be using the standardized vinegar from this experiment in the next experiment to determine the concentration of ammonium hydroxide in household ammonia solution. How do you expect the error from this experiment will affect your results in the next one?
8. Reflect on your pre-lab analysis discussion regarding the green chemistry principles for this experiment. Were your expectations correct? What were the pros and cons of this methodology?

Green Chemistry in Action:
Conductometric Titration of Ammonia with Vinegar

There are many ways to investigate a chemical reaction through the use of an electronic probe to measure changes as the reaction proceeds. A common laboratory technique is known as titration—a process used to determine the volume of a known solution needed to react with a given amount of another solution with an unknown concentration, identity, or both. In a past experiment, you may have run an acid-base titration in which you measured the change in pH. In this experiment, you will be performing a conductivity titration.

Let's take a look at what happens when you titrate a strong acid with a strong base and measure the changes in conductivity. For example, consider the reaction of solutions of HCl and NaOH. The reaction equation, expressed in ionic form follows:

$$H^+(aq) + Cl^-(aq) + Na^+(aq) + OH^-(aq) \rightarrow H_2O(\ell) + Na^+(aq) + Cl^-(aq)$$

As you slowly add an NaOH solution of known molar concentration to a solution of HCl of unknown concentration, the conductivity of the reaction mixture changes. As you see in the equation above, the total amount of ionic species (which help conduct electricity) changes from the reactants to the products. A conductivity probe, placed in the reaction mixture, can measure the number of ions in the reaction mixture as a conductivity value, microsiemens/cm, or µS/cm.

Figure 1 shows a sample plot of a conductivity titration of HCl and NaOH. Initially, the conductivity of the HCl solution is high. As NaOH solution is added, the conductivity decreases. At the equivalence point of the titration, the molar amounts of HCl and NaOH are equal and the conductivity of the reaction mixture is at its lowest point. As NaOH solution is added beyond the equivalence point, the sensor is essentially measuring the conductivity of Na^+ and OH^- ions.

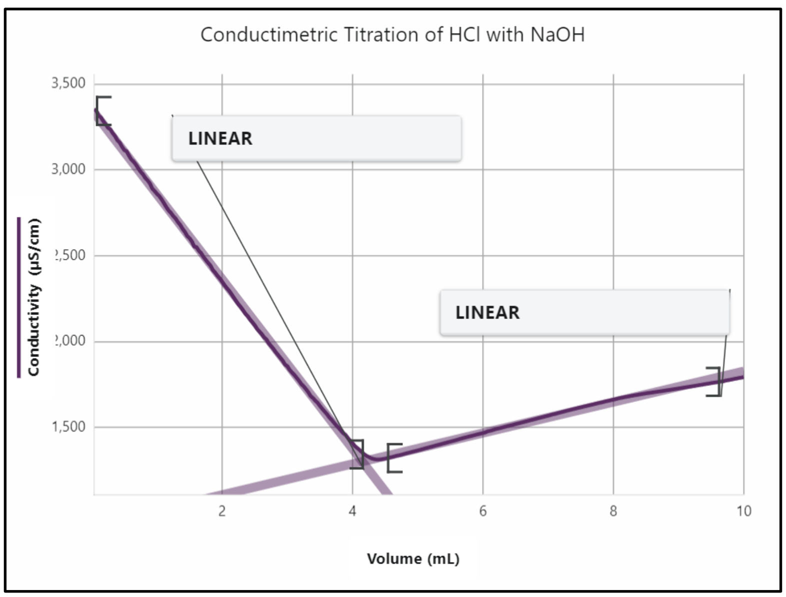

Figure 1

In this experiment, you'll use a conductivity probe to track the progress of the reaction between two common household substances: ammonium hydroxide (ammonia) and acetic acid (vinegar). Your task is to determine how the conductivity of the solution changes as the titration proceeds and to identify the point at which the reaction reaches completion.

By using environmentally benign reagents, this lab not only teaches fundamental chemical concepts but also introduces you to the principles of green chemistry. You will be challenged to think about two important principles of green chemistry: (1) the environmental impact of chemical processes, and (2) the implementation of safer, more sustainable practices in the laboratory.

What changes will you observe? How will the conductivity data inform your understanding of the reaction? How strong are common household chemicals? Can we use the household chemicals to determine the strength of other solutions? What is a titration? Embark on this investigation to find out.

Pre-Lab Analysis

1. **Chemical Reaction and Calculations**
    - Write a balanced chemical equation for the reaction of acetic acid (in vinegar) with store-bought ammonia (ammonium hydroxide).
    - The typical concentration of store-bought ammonia is 5% by weight. Calculate its theoretical molarity.
    - How do you expect these theoretical values to compare with actual values you will measure? How will you compare them?
2. **Observation and Measurement**
    - You will be using a conductivity sensor and live data collection to monitor your titration reaction. Think about the important observations you will need to make to confirm the reaction is complete.
    - We will use a conductivity probe to measure the conductivity of the solution as the titration progresses, how do you think the conductivity will change?
3. **Green Chemistry Application**
    - Consider how using vinegar and ammonia aligns with the principles of green chemistry. Why are these reagents considered less hazardous and more environmentally friendly?
    - Discuss how you can minimize waste and environmental impact during the experiment, including how the use of data-collection technology influences the titration and how you can ensure that all the materials used are biodegradable and safe for the environment.

Materials

- computer, Chromebook™, or mobile device
- Vernier Graphical Analysis® app
- Go Direct® Conductivity (GDX-CON)
- Go Direct Drop Counter (GDX-DC)
- Stir Station and magnetic stirring bar
- test tube clamp
- (2) 250 mL beakers
- 100 mL graduated cylinder
- 10 mL pipet and pump
- commercial vinegar solution of known concentration
- commercial ammonia solution
- wash bottle with distilled water

Investigation

Set up for data collection

1. Launch Graphical Analysis. Connect your Go Direct Conductivity Probe and Go Direct Drop Counter.
2. Prepare the Drop Counter for data collection.
    a. Mount the Drop Counter to the Stir Station as shown in Figure 2.
    b. Click or tap the Volume meter, and choose Calibrate.
        - If the calibration value (drops/mL) is unknown, follow the onscreen instructions under Automatic to calibrate your Drop Counter. You will need a 10 mL graduated cylinder.
        - If the calibration value (drops/mL) is known, click or tap **Manual** and enter the value.

    c. Click or tap **Apply**.

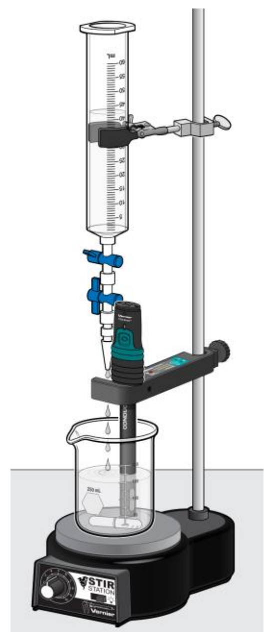

Figure 2

Prepare for the titration

3. Fill the Drop Counter reservoir with vinegar solution of known concentration.

4. Align the Drop Counter reservoir with the opening of the drop counter. Test the drops over a 250 mL beaker centered on the Stir Station.

5. Adjust the drop rate of the Drop Counter to about 1 drop every 2 seconds. Use the top valve to adjust the drop rate and the bottom valve to stop and start the drip.

6. Remove the 250 mL beaker. Then, place a clean 250 mL beaker on the Stir Station and add the magnet to the beaker.

7. Pipet precisely 5.0 mL of ammonia solution into the beaker.

8. Add 100 mL of distilled water to the beaker.

9. Place the conductivity probe in the Drop Counter. Adjust the beaker as needed to make sure the magnet doesn't strike the probe.

10. Set the Stir Station to a speed that is stirring briskly (medium to high setting), but that doesn't create splashing.

Collect titration data

11. Collect data.
    a. Start data collection. No data will be collected until the first drop goes through the Drop Counter slot.
    b. Fully open the bottom valve—the top valve should still be adjusted so drops are released at a rate of about 1 drop every 2 seconds.
    c. When the first drop passes through the Drop Counter slot, check the graph or table to see that the first data pair was recorded.
    d. Stop data collection approximately 3 mL past the inflection point.

12. Dispose of the beaker contents and reset for a second trial. Start data collection when you are ready. **Note**: The first data set is automatically stored.

Post-Lab Analysis

1. Using your titration data, determine the precise volume when the vinegar completely neutralizes the ammonia. Explain the method you used.

2. Calculate the concentration of the ammonia solution (ammonium hydroxide). The known concentration of vinegar may have been determined in a previous experiment. If not, your instructor will give you the known concentration of the vinegar solution.

3. Based on the shape of the conductivity versus volume graph, describe what is happening in the chemical reaction at each of the following stages: (1) the initial change in conductivity, (2) the peak or plateau phase, and (3) the final change in conductivity. Explain how the changes in conductivity relate to the reactions occurring between acetic acid and ammonium hydroxide.

4. Reflect on your pre-lab analysis discussion regarding the green chemistry principles for this experiment. Were your expectations correct? What were the pros and cons of this methodology?