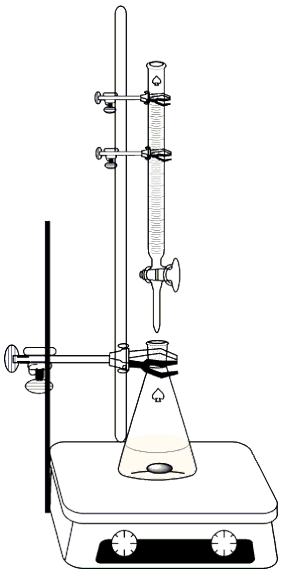
Lab 4.11
Iodometric Titration of Commercial Bleach

AP Chemistry

BACKGROUND



Titration is a technique used to determine the amount of substance present in an unknown sample. A solution of known concentration (the titrant) is added to an unknown substance (the analyte) until a visible endpoint to the reaction is observed. While acid-base titrations use a pH indicator to provide a visible endpoint, a redox reaction relies on the production (or consumption) of a colored ion to provide a visible endpoint. Using the concentration and quantity of the titrant with the quantity of the analyte, the concentration of the analyte may be determined through stoichiometry.

Iodometric titration is a volumetric analysis technique that uses iodine and its oxidation/reduction as an indicator mechanism. Iodine readily reacts with many organic and inorganic substances. Iodometry can be used both to determine amount of reducing agents (by direct titration with iodine) and of oxidizing agents (by titration of iodine with thiosulfate). End-point determination is based on the presence of a blue starch complex.

Many commercial products, particularly cleaning products like bleach, cleansing powders, pool shock, toilet cleaners, etc., contain oxidizing agents. A common oxidizing agent in these products is sodium hypochlorite (NaClO or NaOCl).

Commercial bleaches are made by bubbling chlorine gas into sodium hydroxide solution. Some of the chlorine is oxidized to the hypochlorite ion, ClO^-, and some is reduced to the chloride ion, Cl^-. The solution remains strongly basic. The chemical equation for the process is:

$$Cl_2(g) + 2OH^-(aq) \rightarrow ClO^-(aq) + Cl^-(aq) + H_2O(l)$$

The amount of hypochlorite ion present in a solution of bleach can be determined by an oxidation-reduction titration using iodine and thiosulfate ions. The iodide will be oxidized [$2I^-(aq) \rightarrow I_2(aq) + 2e^-$] due to its low reduction potential relative to other oxidizing agents. In acidic solution, hypochlorite ions oxidize iodide ions to form iodine, I_2. The iodine that forms is then titrated with a standard solution of sodium thiosulfate.

The analysis takes place in a series of steps:

(1) Acidified iodide ion is added to hypochlorite ion solution, and the iodide is oxidized to iodine.

$$2H^+(aq) + ClO^-(aq) + 2I^-(aq) \rightarrow Cl^-(aq) + I_2(aq) + H_2O(l)$$

(2) Iodine is only slightly soluble in water. It dissolves very well in an aqueous solution of iodide ion, in which it forms a complex ion called the triiodide ion. Triiodide is a combination of a neutral I_2 molecule with an I^- ion. The triiodide ion is yellow in dilute solution, and dark red-brown when concentrated.

$$I_2(aq) + I^-(aq) \rightarrow I_3^-(aq)$$

(3) The triiodide is titrated with a standard solution of thiosulfate ions, which reduces the iodine back to iodide ions:

$$I_3^-(aq) + 2S_2O_3^{2-}(aq) \rightarrow 3I^-(aq) + S_4O_6^{2-}(aq)$$

During this last reaction the red-brown color of the triiodide ion fades to yellow and then to the clear color of the iodide ion. It is possible to use the disappearance of the color of the I_3^- ion as the method of determining the end-point, but this is not a very sensitive procedure. Addition of starch to a solution that contains iodine or triiodide ion forms a reversible blue complex. The disappearance of this blue colored complex is a much more sensitive method of determining the end-point. However, if the starch is added to a solution which contains a great deal of iodine, the complex which forms may not be reversible. Therefore, the starch is not added until shortly before the end-point is reached. The quantity of thiosulfate used in step (3) is directly related to the amount of hypochlorite initially present.

Safety

- Both the concentrated bleach and hydrochloric acid are damaging to skins, eyes and clothing.
- Avoid breathing the vapors from the bleach and acid as they are irritants to the respiratory system.
- The addition of hydrochloric acid to bleach may produce chlorine gas in Part B, Step 5, so this step should be completed in a fume hood.
- Spills should be neutralized and cleaned up immediately. Any solutions that contact skin should be rinsed off with plenty of water.
- Goggles and aprons must be worn.

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I. PURPOSE

To test/verify the percent of sodium hypochlorite present in a commercial bleach using a redox titration method.

II. MATERIALS

1. Bleach, NaClO, commercial
2. Starch solution, 2%
3. Hydrochloric acid, HCl, 3M
4. Sodium thiosulfate solution, $Na_2S_2O_3$, 0.100*M*
5. Potassium iodide, KI(s)
6. Balance
7. Ring stand
8. Buret assembly
9. Buret clamp
10. Volumetric Flask (100mL)
11. 250 mL Erlenmeyer flask
12. Pipets (5 & 25mL)
13. Pipet pump/bulb
14. Magnetic stirrer & bar

III. PROCEDURES

Part A – Preparing the Buret

1. Thoroughly clean the buret assembly and rinse with distilled water. Hang the buret assembly using the buret clamp & ring stand.
2. Rinse the buret with a small amount of 0.100*M* sodium thiosulfate solution.
3. Dispose of the rinse solution down the sink.
4. Fill the buret a little above the 0.00 mL level of the buret with 0.100*M* sodium thiosulfate solution.
5. Drain a small amount of 0.100*M* sodium thiosulfate solution so that all air bubbles are flushed from the buret tip.
6. Record the precise concentration of the sodium thiosulfate solution and the initial buret reading in the data table.

Part B – Preparing the Analyte

1. Transfer 5.00mL of the commercial bleach solution to a 100mL volumetric flask (or 100mL graduated cylinder) and dilute to 100mL using distilled water. Stopper and mix the solution thoroughly.
2. Weigh out approximately 2g of KI(s). This reactant will be in excess and does not require careful measurement.
3. Transfer 25.00mL of the diluted bleach solution from Step 1 into a clean 250mL Erlenmeyer flask using the 25mL pipet.
4. Add approximately 25mL of distilled water to the E-flask and add the KI(s) from Step 2. Mix thoroughly until the KI is dissolved.
5. At the fume hood, add 2mL of 3*M* HCl to the E-flask while swirling the solution. ***Avoid breathing the fumes.*** The solution should turn dark-yellow/brown from the production of I_3^- complex ions in the solution.
6. Place the beaker on a magnetic stirrer and add a stirring bar. Turn on the magnetic stirrer and allow it to stir gently.

Part C – Titration

1. Titrate the analyte with the 0.100*M* $Na_2S_2O_3$ until the iodine color becomes light yellow.
2. Add one full dropper pipet of starch soluiton to the E-flask and . *The solution should turn blue as the starch-iodine complex forms.*
3. Continue the titrating the analyte until a single drop of $Na_2S_2O_3$ solution causes the blue color to disappear. Record the final buret reading.
4. Repeat the titration 2 additional times, preparing a fresh analyte for each trial and recharging the buret as necessary.
5. Dispose of the solutions according to the instructor's directions.

IV. PRE-LAB QUESTIONS

1. Define oxidation and reduction.
2. Write balanced oxidation and reduction half-reactions for equations (1) and (3) in the background section. For each half reaction, identify which substance is oxidized or reduced.
3. In this analysis, an "aliquot", or a diluted fraction of the initial solution is used for the titration. What advantage is there in diluting the original solution for the analysis?
   a. How many 25-mL aliquots can be measured from a 100-mL volumetric flask? Explain.
4. Write the equation for the neutralization of acetic acid with sodium hydroxide.
   a. If 17.5 mL of 0.250M sodium hydroxide solution is required to titrate 25.0mL of acetic acid, what is the molarity of the acetic acid?
   b. Assuming the density of acetic acid is 1.05 g/mL, calculate the percentage by mass of acetic acid in the solution.

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V. DATA & CALCULATIONS

A. DATA

Trial	Initial Buret Reading (mL)	Final Buret Reading (mL)	Total $Na_2S_2O_3$ Used (mL)
1			
2			
3			

Molarity of $Na_2S_2O_3$	

B. CALCULATIONS

1. Using the equations from the Background section, determine the relationship between moles of sodium thiosulfate used and moles of sodium hypochlorite present in the analyte.
2. Calculate the average volume of $Na_2S_2O_3$ required for the titration of 25.00mL of the diluted bleach solution.
3. Use the average volume and molarity of $Na_2S_2O_3$ to determine the molarity of the diluted bleach.
4. From the dilution process used, calculate the molarity of the undiluted commercial bleach.
5. Calculate the percent by mass of NaClO in the commercial bleach. *(Assume that the density of the commercial bleach is 1.08 g/ml.)*
6. Calculate the percent error using the percentage by mass expressed on the label of the bleach container as the accepted value.

VI. POST-LAB QUESTIONS

1. The reaction between triiodide (I_3^-) and thiosulfate ions ($S_2O_3^{2-}$) produces the dithionate ion ($S_4O_6^{2-}$). Determine the oxidation number of sulfur in this ion. Provide an explanation for the oxidation number determined with the understanding that oxidation numbers are almost never fractions.
2. How would each of the following laboratory mistakes affect the calculated value of the percent NaClO in the commercial bleach (too high, too low, no change)? Explain.
    a. In Part B, Step 1, the pipet was rinsed with distilled water immediately before being used to measure the commercial bleach solution.
    b. In Part B, Step 2, 3g of KI was used instead of 2g.
    c. In Part B, Step 5, some of the iodine that formed after the addition of the HCl vaporized from the solution.
3. What is the major source of error in this experiment? Explain.

VII. CONCLUSION