Experiment 8 Determination of the Amount of Sodium Hypochlorite in Commercial Bleach: A Redox Titration

Introduction

Chlorine bleach is used to remove stubborn stains from clothes. Actually, the bleach may not remove the stain from the clothes but instead reacts with the stain to remove its color so that the stain cannot be seen. The reason that bleach is generally not used on colored clothes is that the bleach will also remove the colored dye of the clothes as well as the stain.

The active ingredient in most common commercial bleaches is a compound called sodium hypochlorite, NaOCl. Chlorine bleach is simply an aqueous solution of sodium hypochlorite. The hypochlorite ion, ClO^-, is a strong oxidizing agent that is easily reduced to the chloride ion via the half-reaction:

$$OCl^-_{(aq)} + H_2O_{(l)} + 2\ e^- \longrightarrow Cl^-_{(aq)} + OH^-_{(aq)}$$

Being a strong oxidizing agent, the hypochlorite ion will cause other substances to be oxidized. For example, when a sample of potassium iodide is added to a solution of sodium hypochlorite, the iodide ion is oxidized to elemental iodine via the half-reaction:

$$2\ I^-_{(aq)} \longrightarrow I_{2(aq)} + 2\ e^-$$

The balanced redox reaction for this reaction is:

$$2\ I^-_{(aq)} + OCl^-_{(aq)} + H_2O_{(l)} \longrightarrow I_{2(aq)} + Cl^-_{(aq)} + OH^-_{(aq)}$$

In this experiment, the amount of sodium hypochlorite contained in a sample of commercial bleach will be determined by first reacting the bleach with an excess of aqueous potassium iodide. Upon mixing, the redox reaction shown above will occur. Since an excess of iodide ion is used, all of the sodium hypochlorite in solution will have been reduced to chloride ion, leaving an amount of unreacted iodide ion remaining in solution and producing an amount of elemental iodine. The amount of hypochlorite ion contained in the bleach may be calculated by determining the amount of elemental iodine produced. According to the stoichiometric relationship given by the redox reaction above:

$$\text{Number of moles of } OCl^- \text{ in bleach sample} = \text{Number of moles of } I_2 \text{ produced}$$

The number of moles of I_2 produced in the reaction mixture will be determined by performing a **redox titration** of the iodine with a sodium thiosulfate solution. The thiosulfate ion, $S_2O_3^{2-}$, reacts with elemental iodine via the following redox reaction:

$$I_{2(aq)} + 2\ S_2O_3^{2-}{}_{(aq)} \longrightarrow 2\ I^-_{(aq)} + S_4O_6^{2-}{}_{(aq)}$$

A redox titration is performed very similarly to an acid-base titration. Instead of performing an acid-base neutralization reaction, a redox titration uses a redox reaction when the two solutions are added. At the endpoint of this titration, the following stoichiometric relationship will hold according to the redox reaction above:

$$\text{moles of } S_2O_3^{2-} \text{ added} = 2 \times \text{moles of } I_2 \text{ produced}$$

In acid-base titrations, an indicator was added to the solution that changed color near the equivalence point of the titration. Redox titrations have their own indicators that change color near the equivalence point. In this titration, the equivalence point will be found by adding starch to the solution containing the elemental iodine. Starch forms a complex with elemental iodine that has a very dark blue color. As thiosulfate ion is added to the solution, the elemental iodine is removed from the solution via the redox reaction. Adding sodium thiosulfate solution to the iodine solution until the blue color of the iodine-starch complex disappears will indicate the endpoint of the titration. At this point, the number of moles of I_2 contained in solution is twice times the number of moles of $S_2O_3^{2-}$ added. The number of moles of $S_2O_3^{2-}$ added will be equal to the volume of sodium thiosulfate solution added (in liters) times the concentration of the sodium thiosulfate solution.

The concentration of the sodium thiosulfate solution will also be determined in a **standardization** procedure. Sodium thiosulfate solutions are typically standardized using potassium iodate (KIO_3). The redox reaction for the reaction of iodate ion with thiosulfate ion in acidic solution is:

$$IO_3^-{}_{(aq)} + 6\ S_2O_3^{2-}{}_{(aq)} + 6\ H^+{}_{(aq)} \longrightarrow I^-{}_{(aq)} + 3\ S_4O_6^{2-}{}_{(aq)}$$

Therefore, at the endpoint of this titration, the number of moles of $S_2O_3^{2-}$ is related to the number of moles of IO_3^- by the stoichiometric relationship:

$$\text{Moles of } S_2O_3^{2-} = (\text{Moles of } KIO_3)\left(\frac{6 \text{ mol } S_2O_3^{2-}}{1 \text{ mol } IO_3^-}\right)$$

Pre-Lab Assignment

1. Update table of contents
2. Set up lab notebook for the experiment
    a. Experiment title
    b. Experiment purpose
    c. Brief procedure or flow chart
    d. Construct the necessary data table for the experiment as shown below. If you have questions regarding the data table, please speak with your instructor *before* lab.

Equipment

The following equipment will be needed from your tote box for this experiment:

> 30-mL beaker, 15-mL beaker, 12-mL Erlenmeyer flask, 250-mL Erlenmeyer flask, 10-mL graduated cylinder

The following equipment will be supplied to you in the lab:

> 25-mL volumetric pipette, 100-mL volumetric flask with stopper, burette

When finished, please put the volumetric flasks back from where you got them from. *Do not put them in your tote box!!*

Procedure

Standardization

1. Fill your burette with $Na_2S_2O_3$ solution. There should be a reagent bottle of KIO_3 solution with its concentration written on the bottle. Record the concentration of KIO_3 on your data sheet.
2. Pour a little over 50 mL of the KIO_3 solution into your 150-mL beaker.
3. Obtain a 25-mL volumetric pipette and fill it to the mark with the KIO_3 solution. Rinse the volumetric pipette with a small portion of KIO_3 solution before filling.
4. Transfer the KIO_3 solution from the volumetric pipette to a 125-mL Erlenmeyer flask. Add about two grams of solid potassium iodide (KI) to the Erlenmeyer flask. Swirl the flask to dissolve the KI.
5. Add 10.0 mL of 0.5 *M* H_2SO_4 to the flask and swirl to mix. The solution should turn a dark brownish-yellow color due to the formation of iodine (I_2).
6. Begin the titration with $Na_2S_2O_3$ solution until the color fades to a pale yellow.
7. Add 2 mL of starch solution to the flask at this time. The solution should turn to a dark blue-purple color. Be careful at this step, you should still have a yellow color when you add starch. *If no dark color appears when you add starch, then you have already passed the endpoint and need to start the titration again with a fresh 25.00 mL portion of KIO_3 solution.* If the dark blue-purple color appears, slowly add more NaS_2O_3 solution until the color suddenly disappears.
8. Read the volume of $Na_2S_2O_3$ solution added at this point to the nearest 0.01 mL. Repeat this titration for a second portion of 25.00 mL of KIO_3 solution.

Determination of the Amount of NaOCl in Bleach

1. Fill your burette with $Na_2S_2O_3$ solution.
2. Weigh a 30-mL beaker to the nearest 0.001 g.

3. Obtain a sample of liquid chlorine bleach. Measure out 10.0 mL of the bleach in your 10-mL graduate cylinder.
4. Pour the bleach into the 30-mL beaker and weigh it to the nearest 0.001 g. Determine the mass of bleach used by difference methods.
5. Carefully transfer the bleach into a 100-mL volumetric flask using a funnel.
6. Dilute to the mark of the volumetric flask with deionized water.
7. Cap the flask and mix the solution thoroughly.
8. Measure out 25.00 mL of this solution in a 25-mL volumetric pipette. Rinse the 25-mL volumetric pipette with a little of the diluted bleach solution before filling.
9. Transfer the 25.00 mL portion of the diluted bleach solution into a 250-mL Erlenmeyer flask.
10. Add about 20 mL of deionized water, 2.0 g of KI, and 10.0 mL of H_2SO_4 to the Erlenmeyer and swirl to dissolve. The solution should turn yellow-brown due to formation of iodine.
11. Begin the titration by adding $Na_2S_2O_3$ solution until the color fades to a pale yellow.
12. Add 2 mL of starch solution to the flask. The solution should turn a dark blue-purple color. Continue adding $Na_2S_2O_3$ solution until the color disappears.
13. Read the volume of $Na_2S_2O_3$ solution added to the nearest 0.01 mL.
14. Repeat this procedure for a second 25.00 mL portion of diluted bleach solution.

Calculations

For the standardization procedure, calculate the number of moles of IO_3^- used by multiplying the volume of KIO_3 solution used (25.00 mL or 0.0250 L) times the concentration of KIO_3:

$$\text{Moles of } IO_3^- = (0.02500 \text{ L}) \times (\text{concentration of } KIO_3)$$

Convert the number of moles of IO_3^- into the number of moles of $S_2O_3^{2-}$ by using the stoichiometric relationship in the balanced redox reaction of IO_3^- and $S_2O_3^{2-}$:

$$\text{Moles of } S_2O_3^{2-} = (\text{Moles of } IO_3^-)\left(\frac{6 \text{ mol } S_2O_3^{2-}}{1 \text{ mol } IO_3^-}\right)$$

The concentration of the $Na_2S_2O_3$ solution is calculated by dividing the number of moles of $S_2O_3^{2-}$ by the volume of solution added in liters:

$$[Na_2S_2O_3] = \frac{\text{moles of } S_2O_3^{2-}}{\text{volume of } Na_2S_2O_3 \text{ added in liters}}$$

Average the calculated concentrations for the two standardization procedures to obtain the concentration of the standardized $Na_2S_2O_3$ solution.

For the bleach titrations, calculate the number of moles of $S_2O_3^{2-}$ added in the titration by multiplying the concentration of the standardized $Na_2S_2O_3$ solution by the volume of the solution added in liters:

$$\text{Moles of } S_2O_3^{2-} \text{ added} = (\text{volume of } Na_2S_2O_3 \text{ added in liters}) \times [Na_2S_2O_3]$$

Convert the number of moles of $S_2O_3^{2-}$ added into number of moles of OCl^- by using the stoichiometric relationship in the balanced redox reaction:

$$\text{Moles of } OCl^- = (\text{Moles of } S_2O_3^{2-} \text{ added})\left(\frac{1 \text{ mol } OCl^-}{2 \text{ mol } S_2O_3^{2-}}\right)$$

The number of moles of OCl^- in the titration came from 25.00 mL out of 100.00 mL of the diluted bleach sample. In order to calculate the number of moles of OCl^- in the diluted bleach sample, multiply the number of moles of OCl^- by 4 (= (100 mL/25.00 mL):

$$\text{Moles of } OCl^- \text{ in diluted bleach sample} = \text{Moles of } OCl^- \times 4$$

Calculate the number of grams of NaOCl in your 10.0 mL sample of bleach by multiplying the number of moles of OCl^- in the diluted bleach sample by the molar mass of NaOCl:

$$\text{Grams of NaOCl} = (\text{Moles of } OCl^- \text{ in diluted bleach sample}) \times (\text{molar mass of NaOCl})$$

Calculate the %NaOCl in your bleach sample by applying the following formula:

$$\%\text{NaOCl} = \frac{\text{Grams of NaOCl}}{\text{Mass of bleach}} \times 100$$

Since you have two titrations, you should have two values for %NaOCl. Average the two values to obtain the %NaOCl in the bleach.

Name: ______________________________________ CHEM 1412 Section: _________

Partner: ____ Date: ___/___/___

Report Sheet EXP8: Determination of the Amount of Sodium Hypochlorite in Commercial Bleach: A Redox Titration

Standardization of $Na_2S_2O_3$ solution

Concentration of KIO_3 solution	____________	
Final Burette Reading	____________	____________
Initial Burette Reading	____________	____________
Volume of $Na_2S_2O_3$ solution added:	____________	____________
Moles of IO_3^-	____________	____________

Calculation:

Moles of $S_2O_3^{2-}$ added:	____________	____________

Calculation:

Concentration of $Na_2S_2O_3$ solution	____________	____________

Calculation:

Average concentration of $Na_2S_2O_3$	____________	

Amount of NaOCl in Bleach Sample

Mass of beaker and bleach	____________	
Mass of beaker	____________	
Mass of bleach	____________	
Final burette reading	____________	____________
Initial burette reading	____________	____________
Volume of $Na_2S_2O_3$ solution added	____________	____________
Moles of $S_2O_3^{2-}$ added	____________	____________

Calculation:

Moles of OCl^- neutralized	____________	____________

Calculation:

Moles of OCl^- in bleach sample	____________	____________
Mass of NaOCl in bleach sample	____________	____________

Calculation:

%NaOCl in bleach sample	____________	____________

Calculation:

Average %NaOCl in bleach	____________	

Name: ______________________________ Date: ___/___/___ CHEM1412 Section: ________

Experiment 8: Pre-Lab Exercise

Following the instructions in your lab manual, you have titrated a 25.00 mL sample of 0.0100 M KIO_3 with a solution of $Na_2S_2O_3$ of unknown concentration. The endpoint was observed to occur at 16.50 mL.

1. How many moles of KIO_3 were titrated? ___________________
   Show work!

2. How many moles of $Na_2S_2O_3$ did this require? ___________________
   Show work!

3. What is the concentration of the $Na_2S_2O_3$ solution? ___________________
   Show work!

4. What type of reaction was this? ______________________________