EXPERIMENT 11

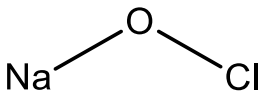
ANALYSIS OF BLEACH BY REDOX TITRATION

Objectives:

In this experiment, the concentration of sodium hypochlorite, the active ingredient in bleach, will be determined through conducting a redox titration. The titration utilizes the reduction of the iodine to form the iodide ion which is correlated to moles of hypochlorite ion in the original bleach sample. The redox titration uses a starch indicator to visualize the endpoint of the titration.

Background:

The active ingredient in commercial bleach products is the strong oxidizing agent sodium hypochlorite, NaOCl. In this procedure, a commercial bleach product is analyzed using titration to determine the mass percent of sodium hypochlorite in the solution.



Sodium hypochlorite is not directly titratable and thus it is first reacted to produce a substance that can be detected through titration, iodine, I_2. This reaction is an **oxidation-reduction reaction** in which the hypochlorite oxidizes iodide ion, I^-, to iodine, I_2, in an acidic solution. Potassium iodide, KI, is added in excess as the source of the iodide ion. Because there is a large excess of I^- in solution, the iodine that is formed during the reaction resides as triiodide ion, I_3^- (I_2-I^-) which is responsible for the dark brown color of the solution. This reaction takes place in a beaker before the titration begins. It is important to allow time for the reaction to complete beore titrating the solution. It should be noted that the product, I_2, is the analyte in the titration.

$$OCl^- + 2I^- + 2H_3O^+ \rightarrow I_2 + Cl^- + 3H_2O$$

The solubility of iodine, I_2, in aqueous solution is relatively low. For this reason the bleach solution cannot be analyzed directly but must be diluted so that the oxidation of the iodide takes place in plenty of solvent. For this purpose, 10 mL of the bleach is diluted to 100 mL in a volumetric flask.

Once formed, the **analyte** iodine is readily titrated with sodium thiosulfate. A standard solution of thiosulfate is prepared in a volumetric flask as the **titrant** using solid sodium thiosulfate. The

stoichiometry of the titration reaction is shown in the equation below. At the equivalence point of the titration the moles of thiosulfate added is twice the number of moles of hypochlorite ion originally present in the titration solution because the molar ratio between the hypochlorite ion and iodine is one to one.

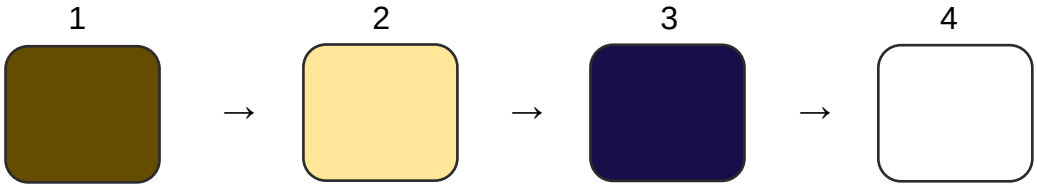
The titration reaction is also an oxidation-reduction reaction where the iodine is reduced back to iodide ion and the sulfur in the thiosulfate ion is oxidized to form the tetrathionate ion.

$$I_2 + 2S_2O_3^{2-} \rightarrow 2I^- + S_4O_6^{2-}$$

The equivalence point of the titration is identified by an endpoint using a starch indicator. As long as triiodide is present the starch will form a dark blue starch/triiodide complex. **Once the iodine has all been reduced back to iodide ion the blue color will disappear.** Due to the intensity of the dark-blue starch/triiodide complex in solution the endpoint can be hard to detect making the titration difficult. To minimize this negative impact the starch indicator is not added until near the end of the titration when visual observation indicates that most of the iodine is reacted.

The methodology for the titration can be summed as:

1. React a dilute bleach sample with potassium iodide in an acidic solution.
2. Add titrant until most of the iodine has been reacted.
3. Add the indicator starch.
4. Titrate the remaining iodine until a colorless solution is achieved at the endpoint.



Tasks to be completed:

1. A solution of sodium thiosulfate is prepared in a 250 mL volumetric flask to be used as the titrant.
2. A 10 mL aliquot of a commercial bleach sample is diluted to 100 mL in a volumetric flask.
3. A redox titration is conducted to determine the concentration of sodium hypochlorite in the bleach sample. Starch is used to visualize the endpoint of the titration.

Experimental Procedure: Students work independently.

I. Preparation of Sodium Thiosulfate Standard Solution

1. Using the analytical balance, accurately mass approximately 4.5 g of sodium thiosulfate pentahydrate. Record the exact mass of the sodium thiosulfate on the Data Sheet.
2. Using deionized water in a wash bottle, transfer the solid into a 250.00 mL volumetric flask. Add additional deionized water to fill the 250 mL volumetric flask approximately half way then swirl the flask until all solid is dissolved.
3. Once the solution is homogeneous, dilute to the mark with additional deionized water and mix by placing a cap on the volumetric flask, inverting then shaking 10 times.

II. Preparation a Solution of Commercial Bleach

1. Obtain 20 mL of stock commercial bleach in a 50 mL beaker. Properly prepare a 10.00 mL pipet by rinsing with a small portion of the bleach solution. **Use a separate waste beaker for the bleach rinse!**
2. Pipet 10.00 mL of the stock bleach into a 100.00 mL volumetric flask. Dilute to the mark with deionized water and mix thoroughly by inverting the flask.
3. Immediately rinse the pipet by **drawing up three-five portions of deionized water into the pipet using a pipet bulb and draining into the bleach waste beaker**.

Dispose excess concentrated bleach in waste hood.

Wash beaker and waste container before continuing.

Do not combine concentrated bleach solution and 3M H_2SO_4.
Poisonous chlorine gas will be evolved!

III. Titrate Diluted Bleach solution with Sodium Thiosulfate

1. Rinse a 50 mL buret with deionized water. Prepare the buret with the sodium thiosulfate standard solution by rinsing with a 20 mL portion of the standard solution. Discard the rinse into a clean waste beaker.
2. Mass approximately 2 g KI in a weigh dish. Set aside.
3. Measure 10.0 mL 3M H_2SO_4 into a **50 mL graduated cylinder**. Set aside.
4. Prepare the rinsed pipet for use in the titration by rinsing with a portion of the diluted bleach solution from the volumetric flask. The diluted bleach waste may be mixed with the sodium thiosulfate waste.

5. Pipet 10.00 mL of the diluted bleach solution into a 250 mL Erlenmeyer flask. Add 50 to 60 mL of deionized water and the massed 2 g sample of KI. Mix well on a magnetic stir plate until the KI is dissolved.
6. Add the 10.0 mL 3 M H_2SO_4 measured in step 3 and mix again for approximately 1 minute until I_2 is fully formed. A red-brown color indicates the presence of free I_2.
7. **TITRATE THE LIBERATED I_2 WITH SODIUM THIOSULFATE UNTIL THE YELLOW COLOR OF I_2 BECOMES SOMEWHAT PALE YELLOW, BUT NOT COLORLESS.** At this point add 2.00 mL of the starch solution from a 10 mL graduated cylinder to form a deep blue I_2 -starch complex.
8. Continue titrating until the deep-blue color disappears signaling the endpoint. Only a FEW DROPS of titrant will be needed to reach the endpoint. Drops of the titrant should be added slowly allowing time for the starch-I_2 complex to react and then for the liberated I_2 to be reduced to I^-.
9. Complete a total of four trials of the titration using 10.00 mL aliquots of diluted bleach solution. Remember an **aliquot** is an exact portion of a solution so the volume of titrant needed to reach the endpoint of the titrations should be reproducible. Multiple titrations are necessary to evaluate precision of the work.

Waste Handling and Clean Up:

1. Dispose of all liquid waste in appropriately marked waste containers.
2. Rinse pipet and buret with deionized water. Set aside for instructor to inspect.
3. Wash 250 mL and 100 mL volumetric flasks and caps with soap and water, rinse with deionized water. Set 250 mL flask aside for instructor to inspect.
4. Wash all other glassware with soap and water, rinse with deionized water.
5. Return buret clamps, stir motors and all other equipment to original location.
6. Verify that balance area is clean and balance doors are shut.
7. Wipe down benchtop area, including sink area, with a damp sponge.

Data Analysis:

I. Determine the Concentration of the Sodium Thiosulfate Standard Solution

1. Using a Periodic Table calculate the molar mass of sodium thiosulfate pentahydrate, $Na_2S_2O_3 \cdot 5H_2O$.

2. Using the mass of sodium thiosulfate pentahydrate used to prepare the standard solution and molar mass calculate the number of moles of sodium thiosulfate in the standard solution.

$$moles\ Na_2S_2O_3 = \frac{mass\ Na_2S_2O_3 \cdot 5H_2O}{molar\ mass\ Na_2S_2O_3 \cdot 5H_2O}$$

3. Calculate the molarity of the standard solution. Remember, the sodium thiosulfate pentahydrate was dissolved and diluted to a total volume of 250.00 mL.

$$Molarity\ Na_2S_2O_3 = \frac{moles\ Na_2S_2O_3}{volume\ of\ solution\ prepared,\ L}$$

II. Determine the Mass Percent of Sodium Hypochlorite in the Commercial Bleach

a. Calculate average volume of sodium thiosulfate titrant needed to reach the endpoint

1. Calculate the average volume of sodium thiosulfate (mL) needed to reach the titration endpoint. Remember to analyze for outliers before taking the average.

2. As a measure of precision, determine the standard deviation (s) for the average volume of sodium thiosulfate needed to reach the endpoint. If only three trails were used to determine the average molarity there will only be three terms in the numerator and the denominator will be (3-1).

$$s = \sqrt{\frac{(mL_{trial\,1} - mL_{average})^2 + (mL_{trial\,2} - mL_{average})^2 + (mL_{trial\,3} - mL_{average})^2 + (mL_{trial\,4} - mL_{average})^2}{4-1}}$$

b. Determine the mass percent sodium hypochlorite in commercial bleach

It is important to keep in mind that 10.00 mL aliquots of diluted bleach are titrated. Calculations must take the volume of the aliquot and the dilution of the original bleach into account

1. Using the average volume of sodium thiosulfate needed to reach the titration endpoint and the molarity of the sodium thiosulfate titrant solution, calculate the moles of sodium thiosulfate at the endpoint.

$$(avg\ volume\ Na_2S_2O_3, mL)\left(\frac{1\ L}{1000\ mL}\right)\left(molarity\ Na_2S_2O_3\frac{moles}{L}\right) = moles\ Na_2S_2O_3$$

2. Relate moles of sodium thiosulfate to moles of hypochlorite in the 10.00 mL aliquot.

$$(moles\ Na_2S_2O_3)\left(\frac{1\ mole\ I_2}{2\ moles\ Na_2S_2O_3}\right)\left(\frac{1\ mole\ NaOCl}{1\ mole\ I_2}\right) = moles\ NaOCl\ in\ 10\ mL\ aliquot$$

3. Determine the mass of NaOCl in the prepared 100.00 mL of diluted bleach.

$$\left(\frac{moles\ NaOCl\ in\ aliquot}{10.00\ mL}\right)(100.00\ mL) = moles\ NaOCl\ in\ diluted\ bleach$$

$$(moles\ NaOCl\ in\ diluted\ bleach)\left(molar\ mass\ NaOCl, \frac{g}{mole}\right) = mass\ of\ NaOCl\ in\ bleach\ sample$$

4. Determine the mass percent sodium hypochlorite in the commercial bleach solution from the mass of NaOCl in the bleach sample and the total mass of the original <u>10.00 mL sample</u> of bleach. Assume the density of the bleach solution is the same as that of water, 1.00 g/ml.

$$(volume\ of\ original\ bleach\ sample, ml)\left(\frac{1.00\ g}{ml}\right) = total\ mass\ of\ bleach\ sample, g$$

$$\frac{mass\ of\ NaOCl\ in\ bleach\ sample, g}{total\ mass\ of\ bleach\ sample, g}\ x\ 100\% = mass\ percent\ NaOCl$$

Data Sheet

Date: _____ Name:_____

CHM124L section _____ Instructor_____

Solution Preparation

Mass of $Na_2S_2O_3 \cdot 5H_2O$ _____

Volume of sodium thiosulfate titrant solution prepared _____

Volume of diluted bleach solution prepared _____

Titration of 10.00 mL aliquots of diluted bleach

	Trial 1	Trial 2	Trial 3	Trial 4
Initial buret reading, mL				
Final buret reading, mL				
Volume of $Na_2S_2O_3$ needed to reach endpoint, mL				
Average volume $Na_2S_2O_3$, mL				

Evaluate the precision (repeatability) of your titrations by calculating the standard deviation of the volume of titrant needed to reach the endpoint.

Lab Report

Date: _____ Name:_____

CHM124L section _____ Instructor_____

Determine the concentration of the sodium thiosulfate solution. (Show work)

ANALYSIS	
Molar mass of sodium thiosulfate pentahydrate, $Na_2S_2O_3 \cdot 5H_20$	
Moles of sodium thiosulfate pentahydrate	
Molarity of 250.00 mL solution of sodium thiosulfate	

Lab Report

Determine the mass percent of NaClO in the original bleach solution. (Show all work)

ANALYSIS	
Moles $Na_2S_2O_3$ from average volume	
Moles NaOCl in 10.00 mL aliquot	
Moles NaOCl in diluted bleach	
Mass NaOCl in diluted bleach	
Total mass of 10.00 mL bleach sample	
Mass % NaOCl in original bleach	