

# ANALYSIS OF COMMERCIAL BLEACH

To determine the amount of **sodium hypochlorite (NaClO)** in commercial bleach by means of an oxidation-reduction “back-titration”.

Some commercial products that contain oxidizing agents are bleaches, hair coloring agents, scouring powders, and toilet bowl cleaners. The most common oxidizing agent in bleaches is **NaClO**. Commercial bleach is made by bubbling **chlorine gas** into a **sodium hydroxide** solution. Some of the chlorine oxidizes to the **hypochlorite ion ClO<sup>-</sup>**, and some reduces to the **chloride ion Cl<sup>-</sup>**. The solution remains strongly basic. The **net ionic equation** for this process is:



The hypochlorite ion **ClO<sup>-</sup>** contains the **chlorine atom** in the **+1** oxidation state. Since the **chloride ion Cl<sup>-</sup>** is much more stable (less potential energy) than the **Cl<sup>+1</sup> atom** in the hypochlorite ion, the **Cl<sup>+1</sup>** readily reduces to **Cl<sup>-1</sup>** and thus serves to “oxidize” other substances during the bleaching process.

The amount of **hypochlorite ion** present in a bleach solution is determined by an oxidation-reduction titration called a “back-titration”. While the **ClO<sup>-</sup>** ion cannot be titrated directly, it easily oxidizes the iodide ion **I<sup>-</sup>** in **KI** to form iodine **I<sub>2</sub>** which is then titrated with a standard solution of **sodium thiosulfate Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>**.

The **analysis** takes place in a series of steps:

- 1) **Acidified iodide ion** is added to **hypochlorite ion** solution, and iodide oxidizes to iodine.



- 2) **Iodine**, only slightly soluble in water, dissolves very well in an aqueous solution of iodide ions, in which it forms a **colored complex ion** called the triiodide ion **I<sub>3</sub><sup>-</sup>**. Triiodide ion is a combination of a neutral **I<sub>2</sub>** molecule with an **I<sup>-</sup>** ion. The **I<sub>3</sub><sup>-</sup>(aq)** ion is yellow in dilute solution, and dark red-brown when concentrated.



- 3) The **triiodide ion** is titrated with a standard solution of thiosulfate ions, which reduces the iodine back to the colorless iodide ions.



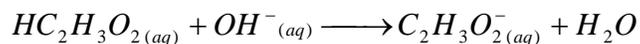
During the last reaction, the red-brown color of the triiodide ion fades to yellow and then to the colorless iodide ion. While it is possible to use the disappearance of the color of the **I<sub>3</sub><sup>-</sup>(aq)** ion as the method of determining endpoint, this is not a very sensitive procedure. **Addition of starch to a solution containing iodine** (or triiodide ion) **forms a reversible blue complex**. The **disappearance of this blue complex** is a much more sensitive method of determining endpoint. However, if the starch is added to a great deal of iodine, the complex which forms may not be reversible. Therefore, the starch is not added until shortly before the endpoint is reached. The quantity of thiosulfate used in step (3) is directly related to the amount of hypochlorite initially present. The **S<sub>4</sub>O<sub>6</sub><sup>2-</sup>** ion is called the tetrathionate ion.

## PRE-LAB ASSIGNMENT

1. Reaction **(1)** above is considered a disproportionation reaction. Research and explain the meaning of this term and explain how it applies to reaction **(1)**

2. Iodine ( $I_2$ ) has very low solubility in water. Explain why this is the case.

3. A solution of household vinegar (a mixture of acetic acid and water) is to be analyzed. A pipet is used to measure out 10.0 mL of the vinegar, which is placed in a 250-mL volumetric flask. Distilled water is added until the total volume of solution is 250 mL. A 25.0-mL portion of the diluted solution is measured out with a pipet and titrated with a standard solution of sodium hydroxide. The neutralization reaction is as follows:



It is found that 16.7 mL of 0.0500 M NaOH is needed to titrate 25.0 mL of the diluted vinegar.

Calculate the molarity of the diluted vinegar.

4. Calculate the molarity of the household vinegar

5. The household vinegar has a density of 1.05 g/mL. Calculate the percent by mass of the acetic acid in household vinegar

## EQUIPMENT

(2) 10mL transfer pipets	50mL buret
(1) 25 mL transfer pipet	buret clamp
(2) 250mL Erlenmeyer flasks	ring stand
25mL graduated cylinder	electronic balance
100.0mL volumetric, cap	glove
(2) 100mL beakers (waste, titrant)	microspatula

## CHEMICALS

bleach, 6.0% NaClO	0.100M Na <sub>2</sub> S <sub>2</sub> O <sub>3(aq)</sub>
starch solution, 2%	KI <sub>(s)</sub>
3M HCl <sub>(aq)</sub>	distilled water

## PROCEDURE

1. Use a pipet bulb and a **10mL transfer pipet** to transfer **5.00mL of commercial bleach** into a **100.0mL volumetric flask**. Dilute to the mark with distilled water, stopper and mix well. Rinse pipet with distilled water three times to wash it.
2. With a **microspatula**, weigh about **2 g KI** into each of 3 small plastic cups on an electronic balance.
3. Use a pipet bulb and a **25ml transfer pipet** to transfer **25.00mL of dilute bleach** into **two 250mL Erlenmeyer flasks**. Add the **2 g KI** to each of the 2 flasks, and about **25mL distilled water**, using a **25mL graduated cylinder**. Swirl to dissolve the KI.
4. In **fume hood**, with **pipet bulb** and **10mL transfer pipet**, add **2mL of 3M HCl** to each flask. The solution should be dark yellow to red-brown from the presence of the **I<sub>3</sub><sup>-</sup>(aq)** complex ions.
5. Fill the **titrant beaker** with about **80mL of 0.100M Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>** and **rinse the buret** twice with about **5mL of titrant**, down the sink. (use a glove) Place **waste beaker** under buret, and fill the buret with titrant, expelling any bubbles from the buret tip, and **fill to the 0.0mL** mark at the top. Record **initial volume** on Data Table.
6. Titrate with the **0.100M Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution** until the iodine color becomes **light yellow**. Add one dropperful of **starch solution**. The **blue color** of the starch-iodine complex should appear. Continue titrating carefully, **drop by drop**, until one drop causes the blue color to disappear. Record **final volume** on Data Table.
7. Repeat step #6 with the other flask.

<b>1<sup>st</sup> Titration mL Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub></b>	<b>2<sup>nd</sup> Titration mL Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub></b>
A.Initial reading _____.	A.Initial reading _____.
B.Final reading _____.	B.Final reading _____.
C.B minus A _____.	C.B minus A _____.

8. The solutions may be safely rinsed down the sink with running water.

## DATA and CALCULATIONS

1. Use the 3 equations given to determine the number of moles of **sodium thiosulfate** that are equivalent to one mole of **sodium hypochlorite**.
2. Calculate the **average volume** of **Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>** needed for the titration of the 3 samples of 25.00mL of diluted bleach.
3. Use the **average volume** (converted to **Liters**) and the given **Molarity** of **Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>** **titrant** to determine the number of **moles** of **Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>** titrant used. ( $M \times V_L = \text{moles}$ )
4. Using the mole-to-mole ratio found in #1 above, convert moles of **Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>** titrant to moles of **NaClO**.
5. Now using the moles of **NaClO** and the **25.00mL volume** (converted to **Liters**) of dilute bleach, calculate the **Molarity** of **NaClO** in the dilute bleach. ( $M \times V_L = \text{moles}$ )
6. Now using the dilution formula: **Molarity<sub>conc</sub> x Volume<sub>conc</sub> = Molarity<sub>dil</sub> x Volume<sub>dil</sub>**  
calculate the **Molarity of the commercial bleach** (concentrated).  
[Note: you diluted **5.00ml** of commercial bleach to **100.0ml** in a flask initially.]
7. From the **Molarity of the commercial bleach**, find **moles** of **NaClO** then **grams** of **NaClO** present in the 5.00mL sample you used ( $M \times V_L = \text{moles}$ ) then (mol -->g)
8. Assume that the **density of the commercial bleach** is **1.08g/ml**, calculate the **mass of the bleach solution** (5.00ml sample) that you used initially
9. Find **Mass%** of the bleach, using your values from step #7 and step #8.
10. If the bleach label reads **6.0% NaClO**, calculate **% Error** of your results.

## DISCUSSION

1. In this analysis, an "aliquot", or a diluted fraction of the initial solution is used for the reaction. What advantage is there in diluting the original solution for the analysis?
2. The reaction with thiosulfate ions produces the tetrathionate ion,  $S_4O_6^{2-}$ . Draw a Lewis dot structure for this molecule. Calculate the oxidation number of sulfur in this ion. Do you think that the sulfur atoms in the ion will all have the same oxidation number? What might the oxidation numbers be?
3. 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 step 1, the pipet was rinsed with distilled water immediately before being used to measure the commercial bleach solution.
  - b. 3 grams of KI was used instead of 2.
  - c. Some of the iodine that formed in step 4 evaporated from the solution