

The Determination of Hypochlorite in Bleach

Reading assignment: Chang, Chemistry 10th edition, pages 156-159.

Goals

We will study an example of a redox titration in order to determine the concentration of sodium hypochlorite, the active ingredient in commercial bleach.

Safety Note: Safety glasses are required when performing this experiment

Equipment and Materials

50.00-mL buret, 250-mL volumetric flask, 25.00-mL volumetric pipette, pipette pump, 300-mL Erlenmeyer flask, 10-mL graduated cylinder, 100-mL graduated cylinder, 100-mL beaker, commercial bleach solution (4-6% hypochlorite), 10% potassium iodide solution (KI), 2 M hydrochloric acid solution (HCl), ~0.26 M sodium thiosulfate solution (Na₂S₂O₃), starch solution.

Discussion

An aqueous solution of sodium hypochlorite (NaOCl) is a clear, slightly yellow liquid, and is commonly known as bleach. Aside from its uses as a bleaching agent, sodium hypochlorite solutions are also used as sterilizing agents and in water treatment. Industrial uses include agriculture, food, paper production, and textiles. Sodium hypochlorite is also added to waste water to reduce odors.

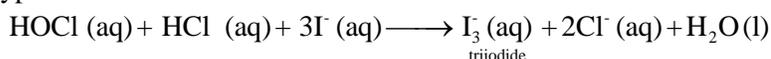
The concentration of sodium hypochlorite in bleach solutions can be determined by titration. A desirable method would be to find a titrant that reacts with NaOCl to form a colored product. But there are no known titrant-indicator systems that work well. Therefore, we must use a multi-step method to titrate sodium hypochlorite.

In the first step sodium hypochlorite, hydrochloric acid, iodide ion, and starch are combined to form a starch-triiodide complex. In this step there are three reactions that take place:

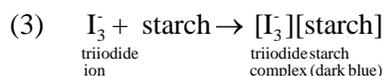
(1) Hydrochloric acid reacts with sodium hypochlorite to form hypochlorous acid:



(2) Hypochlorous acid reacts with iodide when the solution is acidic:

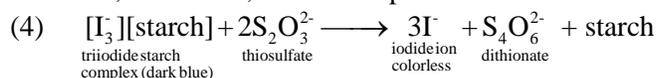


Triiodide, I₃[−], is a dark red complex. A dark blue complex is formed when triiodide is combined with starch.



The result of these three reactions is that when sodium hypochlorite is present the starch-triiodide complex is produced. This is useful because the result of these three reactions is the formation of a dark blue complex that has a concentration that is proportional to the amount of sodium hypochlorite in the solution.

In the next step, the starch-triiodide product is titrated by sodium thiosulfate to form a colorless solution of iodide, dithionate, and uncomplexed starch:



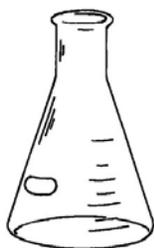
If iodide is added in excess to the hypochlorous acid then all of the hypochlorous acid will be reacted,

forming the dark blue starch-triiodide complex. The hypochlorite acts as a limiting reagent, determining how much triiodide is produced. We can then titrate the triiodide-starch complex with the thiosulfate to determine the concentration of the complex formed. This can then be used to calculate the initial concentration of hypochlorite.

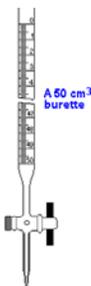
Procedure

SAFETY PRECAUTIONS

Safety glasses or goggles must be worn throughout this experiment. Bleach is a strong oxidizing agent and should be washed from skin. Bleach can also damage clothing. For that reason, take care not to splash any of the bleach solution on clothing.



300 mL Erlenmeyer
Flask



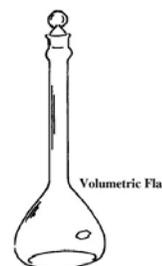
50 mL
buret



25 mL
volumetric pipet



beaker



250 mL volumetric
flask

Students perform titrations individually. Dilution of the bleach (Step 2) can be performed in pairs.

1. Obtain 60 mL of sodium thiosulfate solution using a clean 100-mL beaker. Record the concentration of this solution on the Data Sheet. Rinse a 50-mL buret with tap water and then distilled or deionized water. Rinse the buret with a few milliliters of the sodium thiosulfate solution. Fill the buret to just above the 0 mL mark with sodium thiosulfate solution. Allow a few milliliters to pour through the buret tip so that any trapped air can be flushed through. Read and record the initial buret level to the nearest 0.05 mL.
2. Perform a 10-fold quantitative dilution of bleach. This step can be performed with a partner and the diluted bleach shared. Use a clean 25-mL volumetric pipet to deliver 25.00 mL of bleach solution into a clean 250-mL volumetric flask. Add distilled or deionized water to the mark of the volumetric flask. Be sure to mix the solution well to ensure that it is homogeneous. The bleach should now be one-tenth its original concentration and can be used in your titrations. Rinse the pipet with distilled or deionized water to remove the bleach solution.
3. The titration takes place in a 300-mL Erlenmeyer flask which will be called the reaction flask. Using the 25-mL volumetric pipet, carefully add 25.00 mL of diluted bleach (from step 2) to the reaction flask.
4. Using a 100-mL graduated cylinder, add about 15 mL of distilled or deionized water to the reaction flask.
5. Using a 100-mL graduated cylinder, add about 20 mL of 10% potassium iodide solution to the reaction flask. This 10% KI solution should be clear, and not yellow. If the solution is yellow then it is likely

contaminated.

- Pour about 2 mL of starch solution in a 10-mL graduated cylinder and place the cylinder next to the reaction flask. This starch solution will be added to the flask later.
- Using a 100-mL graduated cylinder, add about 20 mL of hydrochloric acid solution to the reaction flask. Begin the titration promptly after adding this solution to the mixture.
- Begin titrating the bleach solution with sodium thiosulfate solution. Allow about 2–3 mL of the thiosulfate solution to run into the reaction flask and close the stopcock. Don't write the volume down at this point. You'll be adding more thiosulfate in a few moments. There may be a dark precipitate of crystals of solid iodine if you are slow in getting to this stage. It should dissolve and react with the thiosulfate solution by mixing the solution.
- Add the 2 mL of starch solution from the graduated cylinder to the reaction flask. The color of the solution should now be dark blue.
- Continue the titration dropwise with constant swirling until the solution becomes clear. This clear solution signals the endpoint of the titration. Record the buret volume level in the data sheet to the nearest 0.05 mL. The contents of the reaction flask may be washed into the drain.
- Perform two more titrations by repeating steps 3-9.
- After completing your final titration pour any unused reagents down the drain. The buret should be rinsed with distilled or deionized water. All glassware should be returned to the appropriate location.

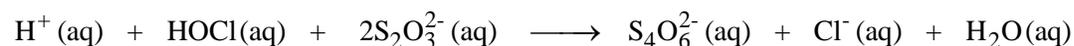
Calculations

To calculate the molarity of the sodium hypochlorite solution we use the titration volume, molarity of the thiosulfate titrant, stoichiometry of the reactions, and volume of the sample of diluted bleach solution.

- Calculate the number of moles of titrant (sodium thiosulfate) for each titration:

$$\text{moles } Na_2S_2O_3 = \left(\text{molarity of } Na_2S_2O_3 \text{ solution} \right) \times \left(\text{volume of } Na_2S_2O_3 \text{ solution} \right)$$

- The moles of hypochlorite are found from the stoichiometry of the reaction with thiosulfate. The stoichiometry of the equation shows that there are two moles of thiosulfate ion per mole of hypochlorous acid. Note that the moles of HOCl are equal to the moles of NaOCl and OCl^- .



$$\text{moles } HOCl = \text{moles } S_2O_3^{2-} \times \left(\frac{1 \text{ mol } HOCl}{2 \text{ mol } S_2O_3^{2-}} \right)$$

- The bleach solution that we titrated was diluted by a factor of 10. Multiply the moles of the hypochlorous acid by 10 to take this factor into account. This will give the number of moles of HOCl in 25.00 mL of undiluted bleach.

4. Sodium hypochlorite (NaOCl) is the form of the hypochlorite that is reported on bleach bottles, not hypochlorous acid. The sodium hypochlorite mass percent is found from the mass of sodium hypochlorite in 25.00 mL and the mass of the 25.00 bleach solution:

$$\text{mass \%} = \left(\frac{\text{mass of NaOCl}}{\text{mass of bleach solution}} \right) \times 100$$

5. The mass of the NaOCl is found from the moles of NaOCl and the molar mass of NaOCl:

$$\text{mass of NaOCl} = (\text{moles of NaOCl}) \times (\text{molar mass of NaOCl})$$

6. The mass of 25.00 mL of bleach solution is found from its density and the volume (25.00 mL):

$$\text{mass of bleach solution} = (\text{density of bleach}) \times (\text{volume of solution})$$

7. Perform a Q test on your mass percent values and calculate an average mass percent concentration. How close is your result to the value listed on the bottle?

N	3	4	5
Q*	0.970	0.829	0.710

$$Q_{calc} = \left| \frac{gap}{range} \right|$$

*95% confidence

Observations and Notes
The Determination of Hypochlorite in Bleach

Date _____

Data Sheet
The Determination of Hypochlorite in Bleach

Name _____

Table 1: Reagent Data

Brand name of bleach _____

Density of bleach _____ $\text{g}\cdot\text{mL}^{-1}$

Volume of diluted bleach used in each titration _____ mL

Concentration of sodium thiosulfate _____ $\text{mol}\cdot\text{L}^{-1}$

Titration Data

	Trial 1	Trial 2	Trial 3
Final buret volume	_____ mL	_____ mL	_____ mL
Initial buret volume	_____ mL	_____ mL	_____ mL
Volume delivered	_____ mL	_____ mL	_____ mL

Calculations

Moles of $\text{Na}_2\text{S}_2\text{O}_3$ in titration	_____	_____	_____
Moles of NaOCl in titration	_____	_____	_____
Moles of undiluted NaOCl	_____	_____	_____
Mass of 25.00 mL of NaOCl	_____ g	_____ g	_____ g
Mass of 25.00 mL bleach solution	_____ g	_____ g	_____ g
Mass % of NaOCl from experiment	_____	_____	_____
Average mass % of NaOCl from experiment (Q test)	_____		
Mass % of NaOCl from label	_____		

