Determination of Sodium Hypochlorite in Household Bleach

Minneapolis Community and Technical College
Principles of Chemistry II, C1152
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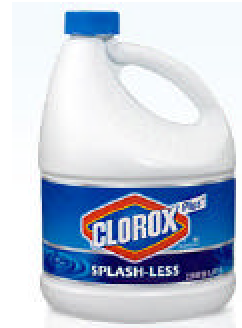

I. Introduction

Chlorine Containing Compounds

The active ingredient in household bleach is sodium hypochlorite, or NaClO. The name of the hypochlorite anion is derived from chemical nomenclature that describes an entire family of chlorine-oxygen containing compounds. This family is represented below:

$$ClO^- \xrightarrow[\text{Energy}]{+1/2\,O_2} ClO_2^- \xrightarrow[\text{Energy}]{+1/2\,O_2} ClO_3^- \xrightarrow[\text{Energy}]{+1/2\,O_2} ClO_4^-$$

hypochlorite — ***chlorite*** — ***chlorate*** — ***perchlorate***

In its ionic form we are accustomed to finding chlorine with a 1- charge. However, each of the chloro-oxo compounds listed above contains chlorine having oxidation states of 1+, 3+, 5+ and 7+ (You should confirm this). These large, positive oxidation states are possible with additional energy that is stored in the compound as chemical potential energy.

The chemical energy stored in chlorate salts can be used in many ways. For example, the solid fuel boosters engines used to propel the space shuttle into orbit contain ammonium perchlorate and powdered aluminum metal. The reaction that occurs as the propellant burns is:

$$3NH_4ClO_{4(s)} + 3Al_{(s)} \rightarrow Al_2O_{3(s)} + AlCl_{3(s)} + 6H_2O_{(g)} + 3NO_{(g)}$$

Chlorine is reduced from its 7+ oxidation state in NH_4ClO_4 to a 1- oxidation state in $AlCl_3$ and Al_2O_3. Aluminum metal is oxidized from its zero oxidation state (Al) to a 3+ oxidation state (Al^{3+}). The reaction releases large amounts of heat energy. The hot, expanding reaction products (hot gases and finely divided solids) are directed out the nozzle of the engine propelling the engine and shuttle upward.

Although not quite as exciting as the perchlorate salts, sodium hypochlorite, the active ingredient in bleach, has interesting chemistry of its own. Household bleach is manufactured by dissolving chlorine gas, Cl_2, in an aqueous solution of sodium hydroxide, NaOH:

Equation 1: $$Cl_{2(aq)} + 2OH^-_{(aq)} \rightleftharpoons ClO^-_{(aq)} + Cl^-_{(aq)} + H_2O_{(l)}$$

This <u>*disproportionation reaction*</u> in which chlorine is both oxidized and reduced, is also an equilibrium reaction signified by the double arrows. The forward reaction (→) converts most of the chlorine gas into hypochlorite and chloride ions. The reverse reaction (←) occurs to a lesser extent and is responsible for producing the chlorine gas that we associate with the smell of bleach.

Mixing bleach with other household cleaning products is a dangerous thing to do. For example, mixing bleach with toilet bowl cleaner (an acid) neutralizes the hydroxide ions in the bleach solution (a base) to form water. Now, according to Le Châtelier's principle, the equilibrium is driven by the reverse reaction (←) to *replace the hydroxide ions* that were neutralized. Cl_2, also produced by the reverse reaction, escapes as a dangerous cloud of gas that can seriously hurt the unsuspecting bathroom cleaner. **Note also that chlorine bleach should never be mixed with ammonia containing compounds. The chloroamine compounds that form are also poisonous.**

Lastly the question: “How does bleach *remove* stains?” The answer is that it doesn’t. A stain absorbs some colors of light and reflects others because of alternating single and double bonds within the stain molecule. The hypochlorite ion, acting as an oxidizing agent, removes electrons from the stain’s double bonds disrupting the alternating single/double bond pattern. Now the stain molecules reflect most visible light rendering the stain “invisible”. Interestingly, in some cases the stain can be still seen under *ultraviolet* light.

Titration of bleach

In this experiment we will determine the amount of sodium hypochlorite that is present in small samples of ordinary household bleach. You are invited to bring along samples of bleach containing cleaning products from home if you like. Be warned that colored cleaning products may mask the color change signifying the endpoint of the titration thus producing less accurate results.

The first step of the bleach analysis reacts sodium hypochlorite with excess iodide and hydrogen ions:

Equation 2: $ClO^-_{(aq)} + H^+_{(aq)} + 2I^-_{(aq)} \rightarrow I_{2(aq)} + Cl^-_{(aq)} + OH^-_{(aq)}$

Iodide ions are added to the reaction mixture by dissolving solid KI. Hydrogen ions are supplied by a small amount of sulfuric acid. The reaction produces only as many I_2 molecules as there were ClO^- ions to begin with.

The iodine molecules that are produced react with additional aqueous iodide ions and starch molecules to produce a blue color (occurs also for standardization trials):

$$\underset{\textbf{colorless}}{starch_{(aq)}} + I^-_{(aq)} + I_{2(aq)} \rightleftharpoons \underset{\textbf{blue}}{I_3^-...\text{starch complex}}$$

Both reactions occur instantly and the blue color is immediately observed.

The actual titration involves the careful addition of aqueous sodium thiosulfate. The thiosulfate ion reacts with I_2 producing iodide ions:

Equation 3: $2S_2O_3^{2-}{}_{(aq)} + I_{2(aq)} \rightleftharpoons 2I^-_{(aq)} + S_4O_6^{2-}{}_{(aq)}$

The effect of this reaction is to remove I_2 from the solution. When all of the I_2 (originally produced via the reaction with hypochlorite) is removed from the solution, the solution changes from blue to colorless (you must have I_2 for the blue starch complex to form).

PRELAB EXCERCISE:

1. **Use equations 2 and 3 (Introduction text above) to determine the mole ratio that relates moles of $S_2O_3^{2-}$ to moles of ClO^-.**

2. **Use equations 4 and 5 (Data analysis section below) to determine the mole ratio that relates moles of IO_3^- to $S_2O_3^{2-}$**

II. MSDS: Chemical Information

Sodium Thiosulfate

CAUTION! MAY BE HARMFUL IF SWALLOWED OR INHALED. MAY CAUSE IRRITATION TO SKIN, EYES, AND RESPIRATORY TRACT.
Health Rating: 0 - None
Flammability Rating: 0 - None
Reactivity Rating: 0 - None
Contact Rating: 1 - Slight
Lab Protective Equip: GOGGLES; LAB COAT
Storage Color Code: Orange (General Storage)
Potential Health Effects
Inhalation: May cause mild irritation to the respiratory tract.
Ingestion: May cause mild irritation to the gastrointestinal tract.
Skin Contact: May cause mild irritation and redness.
Eye Contact: May cause mild irritation, possible reddening.
Chronic Exposure: Chronic exposure may cause skin effects.
Aggravation of Pre-existing Conditions: No information found.

Sodium Hypochlorite

WARNING! HARMFUL IF SWALLOWED OR INHALED. CAUSES IRRITATION TO EYES AND RESPIRATORY TRACT. CAUSES SUBSTANTIAL BUT TEMPORARY EYE INJURY.
Health Rating: 2 - Moderate
Flammability Rating: 0 - None
Reactivity Rating: 1 - Slight
Contact Rating: 2 - Moderate
Lab Protective Equip: GOGGLES; LAB COAT
Storage Color Code: Orange (General Storage)
Potential Health Effects
Inhalation: May cause irritation to the respiratory tract, (nose and throat); symptoms may include coughing and sore throat.
Ingestion: May cause nausea, vomiting.
Skin Contact: May irritate skin.
Eye Contact: Contact may cause severe irritation and damage, especially at higher concentration.
Chronic Exposure: A constant irritant to the eyes and throat. Low potential for sensitization after exaggerated exposure to damaged skin.
Aggravation of Pre-existing Conditions: Persons with impaired respiratory function, or heart disorders (or disease) may be more susceptible to the effects of the substance.

Ammonium Molybdate

Route Of Entry Inds - Inhalation: NO
Skin: YES
Ingestion: YES
Carcinogenicity Inds - NO
Effects of Exposure: AMMONIUM MOLYBDENUM-IRRITANT TO UPPER RESPIRATORY SYSTEM & DIGESTIVE SYSTEM. MAY CAUSE LIVER/KIDNEY DAMAGE.
Explanation Of Carcinogenicity: NONE
Signs And Symptions Of Overexposure: IRRITATION, NAUSEA, DIARRHEA, ANEMIA.
Medical Cond Aggravated By Exposure: PRE-EXISTING EYE PROBLEMS, SKIN DISORDERS, CHRONIC RESPIRATORY DISEASE.
First Aid:
EYES: IRRIGATE IMMEDIATELY W/LARGE QUANTITIES OF UNNING WATER.
SKIN: FLUSH IMMEDIATELY W/WATER. INHALATION: REMOVE TO FRESH AIR. GIVE CPR IF NEEDED. INGESTION: DILUTE IMMEDIATELY W/WATER/MILK. INDUCE VOMITING. OBTAIN MEDICAL ATTENTION IN A LL CASES.

Potassium Iodide

CAUTION! MAY CAUSE IRRITATION TO SKIN, EYES, AND RESPIRATORY TRACT.
Health Rating: 2 - Moderate
Flammability Rating: 0 - None
Reactivity Rating: 1 - Slight
Contact Rating: 2 - Moderate
Lab Protective Equip: GOGGLES; LAB COAT; VENT HOOD; PROPER GLOVES
Storage Color Code: Orange (General Storage)
Potential Health Effects
Inhalation:
May cause irritation to the respiratory tract. Symptoms may include coughing and shortness of breath.
Ingestion: Large oral doses may cause irritation to the gastrointestinal tract.
Skin Contact: May cause irritation with redness and pain.
Eye Contact: May cause irritation, redness and pain.
Chronic Exposure: Chronic ingestion of iodides may produce "iodism," which may be manifested by skin rash, running nose, headache and irritation of mucous membranes. Weakness, anemia, loss of weight, and general depression may

also occur.
Aggravation of Pre-existing Conditions: No information found.

Potassium Iodate

CAUTION! MAY BE HARMFUL IF SWALLOWED. MAY CAUSE IRRITATION TO SKIN, EYES, AND RESPIRATORY TRACT. MAY AFFECT BLOOD, KIDNEYS, CENTRAL NERVOUS SYSTEM. TOXIC EFFECTS MAY BE DELAYED.

Health Rating: 1 - Slight
Flammability Rating: 0 - None
Reactivity Rating: 2 - Moderate
Contact Rating: 1 - Slight
Lab Protective Equip: GOGGLES; LAB COAT
Storage Color Code: Orange (General Storage)
Potential Health Effects
Inhalation: May cause irritation to the respiratory tract. Symptoms may include coughing, sore throat, labored breathing, and chest pain.
Ingestion: Causes irritation to the gastrointestinal tract. Symptoms may include nausea, vomiting and diarrhea.
Skin Contact: May cause irritation with redness and pain.
Eye Contact: May cause irritation, redness and pain.
Chronic Exposure: Animal experiments indicate that potassium iodate can damage kidney and blood cells similar to bromates and chlorates. Central nervous system may be affected.
Aggravation of Pre-existing Conditions: Persons with impaired kidney function may be more susceptible to the effects of the substance.

III. Procedure

You will be working separately today. Everyone will do her or his own experiment and report.

Standardization of Sodium Thiosulfate

In this experiment you will be determining the mass percent of sodium hypochlorite in household bleach via a sodium thiosulfate titration. You will standardize the sodium thiosulfate solution before using it to titrate bleach.

Use an ***analytical balance*** to preweigh a small beaker Using a ***top loading balance*** place approximately 0.5 grams of solid KIO_3 in the beaker. Reweigh using the ***analytical balance*** and determine the precise amount of KIO_3 dispensed to the nearest tenth of a milligram. Use a long stemmed funnel, to transfer the KIO_3 to a 250 mL volumetric flask. Rinse the beaker several times with distilled water transferring each rinse to the volumetric flask. Beware of the tipping hazzard.

Add enough distilled water to the volumetric flask to bring the liquid level (meniscus) up to the line found on the neck of the flask. While holding the glass stopper in the top of the volumetric flask, repeatedly invert the flask and shake until the KIO_3 completely dissolves. Calculate the concentration of the KIO_3 solution you have made .

Use a 10 mL pipette to transfer 10 mL of the KIO_3 solution to a 125 mL Erlenmeyer flask. (Refer to the volumetric pipette operational information available on the chemistry website) Add approximately 13 mL of distilled water to the flask followed by approximately 1 gram of solid KI. Add a magnetic stir bar and stir until all solids have dissolved. Next add 5 mL of 1 M H_2SO_4 and gently stir. The mixture should turn brown due to the presence of I_2 in the mixture.

Rinse and fill a buret with 0.05 M $Na_2S_2O_3$. .

Titrate the KIO_3 mixture (in flask) with the $Na_2S_2O_3$ solution until the color changes to *a very pale yellow..)* Add approximately 3 mL of starch solution to the flask.

The reaction mixture should now be blue signifying the presence of the I3- starch complex Hand swirl the mixture to rinse any solution adhering to the sides of

the flask into the bulk solution. Continue titrating ***CAREFULLY*** until the blue color *disappears*. The endpoint is *very* sharp. Record the final buret reading that corresponds to the endpoint in your notebook.

Repeat the standardization a second time (DO NOT remake the standard solution!)
All waste solutions may be flushed down the sink.

Determination of Sodium Hypochlorite in Bleach

Add approximately 1 gram of solid KI to approximately 25 mL of distilled water in a 125 mL Erlenmeyer flask and swirl to dissolve the KI. Use the analytical balance to determine the mass of the apparatus and record in your lab notebook. Add approximately 0.5 grams of bleach to the reaction flask (approx. 10 drops) Reweigh the apparatus (analytical balance) and determine the mass of the bleach sample to the nearest tenth of a milligram. Place a magnetic stir bar in the flask and stir the mixture on a stir plate until all of the KI has dissolved.

Next, Add 13 mL of 1 M H_2SO_4 and 3 drops of ammonium molybdate catalyst. Gently swirl to mix and set aside for several minutes.

Refill the buret with $Na_2S_2O_3$. Gently stir the mixture in the flask with a magnetic stir bar and titrate the contents with the $Na_2S_2O_3$ solution. When the color has changed from brown to a very pale yellow, add 3 mL of starch indicator solution. Hand swirl the mixture to rinse any solution adhering to the sides of the flask into the bulk solution. Continue titrating slowly until the blue color disappears and record the reading. Record your final buret reading and repeat the experiment using a new sample of **the same bleach.** (Checking for reproducibility).

pH Testing of Old and New Bleach:

Your instructor will demonstrate the pH differences between new and old bleach. Record your detailed observations for this demonstration. Watch carefully as changes occur very quickly.

IV. Data Analysis

1. Calculate the $Na_2S_2O_3$ concentration for each standardization trial using the following equations to obtain the mole ratio that relates $Na_2S_2O_3$ to KIO_3:

Equation 4: $2S_2O_3^{2-}{}_{(aq)} + \mathbf{I_{2(aq)}} \leftrightarrow 2I^-{}_{(aq)} + S_4O_6^{2-}{}_{(aq)}$

Equation 5: $IO_3^-{}_{(aq)} + 5I^-{}_{(aq)} + 6H^+{}_{(aq)} \leftrightarrow \mathbf{3I_{2(aq)}} + 3H_2O_{(aq)}$

2. Calculate the $Na_2S_2O_3$ concentrations for each trial (see data table).If you are confident of both standardization trials, average the $Na_2S_2O_3$ concentrations together. Otherwise pick and use the concentration from the best standardization trial in your bleach calculations.
3. Use the buret readings from the bleach titration and the $Na_2S_2O_3$ concentration from the standardization trials to determine the moles and grams of NaClO in each sample. Chemical reactions required to determine the correct mole ratios are found in the introduction section of this handout.
4. Mass % Sodium hypochlorite is determined as follows:

$$\text{mass }\%_{NaClO} = \text{mass}_{NaClO}/\text{mass}_{bleach} \times 100\%$$

Data Tables:

	Trial 1	Trial 2	*Trial 3
mass of KIO_3 (g)			
total moles of KIO_3			
concentration KIO_3 solution (M)			
volume KIO_3 titrated(mL)			
moles of KIO_3 titrated			
Initial Buret Reading (mL)			
Final Buret Reading (mL)			
Volume $Na_2S_2O_3$ dispensed (mL)			
moles $Na_2S_2O_3$ (calculated)			
Concentration $Na_2S_2O_3$ (M)			
Avg. $Na_2S_2O_3$ molarity (report. with 8 SF)			

	Trial 1	Trial 2	*Trial 3
	Bleach ID	*Bleach ID*	*Bleach ID*
Mass of Bleach used (g)			
Initial Buret Reading (mL)			
Final Buret Reading (mL)			
Volume $Na_2S_2O_3$ dispensed (mL)			
Concentration of $Na_2S_2O_3$ (use avg. value)			
moles $Na_2S_2O_3$			
moles I_2			
moles ClO^- reacted			
moles NaClO originally present			
Mass NaClO originally present (g)			
Mass % NaClO			
Average Mass % (use best two trials)			

* 3rd trial optional

V. Individual Report

Page 1:

- **Upper right corner:** Your name, Your lab section number, Date of experiment
- **Data tables :**
  Obtain a copy of the data table from the lab-handout web site and fill in your values.

Answers to the following questions:

1. What did you observe when you initially exposed the pH paper to new bleach? Did this confirm the bleach was acidic? ...basic?

2. How did the appearance of the pH paper change in the first few seconds of exposure to new bleach? How do you account for the changes?

3. How will leaving the cap off a bleach bottle affect its potency? Use the chemical equilibrium equation provided on page 1 in your answer.

4. Typically, the NaClO mass percent in a fresh bottle of bleach is approximately 5.25%. How does your bleach's mass % compare? Does this comparison suggest anything about the age of the bleach solution you tested? (SHOW ALL WORK)