Determination of Sodium Hypochlorite in Household Bleach

Minneapolis Community and Technical College
Principles of Chemistry II, C1152
v.12.15

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:

	Energy		***Energy***		***Energy***		
ClO^-	$+1/2\ O_2$ -- >	ClO_2^-	$+1/2\ O_2$ -- >	ClO_3^-	$+1/2\ O_2$ -- >	ClO_4^-	
hypochlorite		***chlorite***		***chlorate***		***perchlorate***	

In its ionic form we are accustomed to finding chlorine with a 1- oxidation state. 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 once used to propel the space shuttle into orbit contained ammonium perchlorate and powdered aluminum metal. The reaction that occurred as the propellant burned is:

$3NH_4ClO_{4(s)}$	+	$3Al_{(s)}$	--- >	$Al_2O_{3(s)}$	+	$AlCl_{3(s)}$	+	$6H_2O_{(g)}$	$+3NO_{(g)}$
+7		0		+3 -2		+3 -1			

Chlorine is reduced (gains electrons) from its 7+ oxidation state in NH_4ClO_4 to a 1- oxidation state in $AlCl_3$. Aluminum metal is oxidized (loses electrons) from its zero oxidation state (Al) to a 3+ oxidation state (Al^{3+}). The reaction releases large amounts of heat energy and 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 (Sodium is a spectator ion):

$Cl_{2(aq)}$	+	$2OH^-_{(aq)}$	*forward* < ---- > *reverse*	$ClO^-_{(aq)}$	+	$Cl^-_{(aq)}$	+	$H_2O_{(l)}$

This ***disproportionation reaction*** 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. Equilibrium is achieved when the rate of the forward reaction equals the rate of the reverse reaction.

As you probably are aware, opening a bottle of bleach releases chlorine gas; something we can detect with our noses. As the chlorine gas escapes into the air, the reaction above replaces the lost chlorine by converting products into reactants via the reverse reaction. This uses up the supply of available ClO- in

solution and reduces thus reduces the potency of the bleach. If left uncapped for a long period of time, all of the chlorine will escape and the bleach will be worthless.

So 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.

Important warning: 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. However, Cl_2 is also produced by the reverse reaction and 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 cleaners as the chloroamine compounds that form are also poisonous.**

Standardization of $Na_2S_2O_3$ solution.

You will be titrating your bleach sample with a 0.05 molar solution of $Na_2S_2O_3$ solution (sodium thiosulfate). This solution will fill your burette and be carefully dispensed until the desired endpoint is reached. Initial and final burette measurements are used along with the solution's concentration to determine the number of moles of $Na_2S_2O_3$ used. (i.e. moles = molarity x Liters)

However, the bottle label concentration for $Na_2S_2O_3$ (0.05 M) isn't accurate enough for our purposes and we'll improve on it using a technique called "standardization."

To standardize the $Na_2S_2O_3$ solution, we'll rely on several accurate measurements performed throughout the procedure. The first is a KIO_3 mass measurement performed on the analytical balance with 4 significant figure accuracy. The second are our burette measurements that must ALWAYS be recorded with 2 decimal place accuracy. As long as we perform these measurements with maximum accuracy, we can report the $Na_2S_2O_3$ concentration with 4 significant figures. Your result should be close to 0.05 M but have four significant figures.

The following two sequential reactions take place during the standardization procedure and relate moles of KIO_3 to moles of $Na_2S_2O_3$. Note that potassium and sodium are spectator ions and therefore don't appear.

Reaction #1: $2S_2O_3^{2-}{}_{(aq)} + \mathbf{I_{2(aq)}} \leftrightarrow 2I^-{}_{(aq)} + S_4O_6^{2-}{}_{(aq)}$

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

Titration of bleach

We will determine the amount of sodium hypochlorite that is present in small samples of ordinary household bleach and 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:

Reaction #3: $\mathbf{ClO^-_{(aq)} + H^+_{(aq)} + 2I^-_{(aq)} \text{ -- > } I_{2(aq)} + Cl^-_{(aq)} + OH^-_{(aq)}}$

Excess iodide ions are added to the reaction mixture by dissolving solid KI. Excess hydrogen ions are supplied by a small amount of sulfuric acid solution. The reaction converts ClO^- into I_2 in a 1:1 mole ratio.

The iodine produced above then reacts with excess iodide ions and starch molecules to produce a blue color due to the I_3^- product species according to the following reaction:

$$\text{starch}_{(aq)} + I^-_{(aq)} + I_{2(aq)} \text{ < --- > } I_3^-\text{...starch complex}$$

blue

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

The actual titration involves the careful addition of aqueous sodium thiosulfate using a burette. When added to the mixture, the thiosulfate ion reacts with I_2 producing iodide ions:

Reaction #4: $2S_2O_3^{2-}{}_{(aq)} + I_{2(aq)} \text{ < --- > } 2I^-_{(aq)} + S_4O_6^{2-}{}_{(aq)}$

This reaction removes I_2 from the solution and when it's all gone, the solution changes from blue to colorless endpoint signifying the end of the titration.

II. PRELAB EXERCISE

Clearly answer these questions in INK in your lab notebook before coming to lab.

1. **Use dimensional analysis and reactions 1 & 2 to determine the mole ratio that relates moles of IO_3^- to $S_2O_3^{2-}$. *(Hint: there are only two mole ratios to consider)***

2. **Use dimensional analysis and reactions 3 & 4 to determine the mole ratio that relates moles of $S_2O_3^{2-}$ to moles of ClO^-. *(Hint: there are only two mole ratios to consider)***

3. **A damaged bottle label has made life difficult for a friendly chemist. The bottle contains 325 mL of an NaOH solution but the concentration is no longer readable.**

The chemist removes 5.00 mL of the NaOH solution using a volumetric pipette. Then she adds water and titrates the resulting solution with hydrochloric acid of known concentration. From this she determines that there were 0.0113 moles of NaOH in the 5.00 mL sample.

a. How many moles of NaOH are there in the original 325 mL solution?

b. What is the concentration of the original NaOH solution?

III. Word Processed Report (1 page)

Page 1

Upper Right Corner:

Name _____
Section number _____
Experimental Date _____

Data Tables: Obtain data table templates from lab website and type in your values and results.

Page 2

Provide example hand written calculations for your standardization and bleach titration procedures.

IV. Procedure

You will be working a partner today.

Standardization of Sodium Thiosulfate

Use an ***analytical balance*** to pre-weigh a small beaker Using a ***top loading balance*** place approximately 0.5 grams of solid KIO_3 in a 50 mL beaker.

Reweigh the beaker & solid on the same *analytical balance* to determine the 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. Be careful not to tip the funnel & flask over as they will break.

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. You will need to remove the funnel from the flask and use an eye dropper for the final water additions.

Insert the stopper into the flask and hold it in place whilst repeatedly inverting the flask and shaking. Continue until the KIO_3 completely dissolves. Calculate the concentration of the KIO_3 solution you have made with excess significant figures.

Use a 10 mL pipette to transfer 10 mL of the KIO_3 solution you've just prepared to a 125 mL Erlenmeyer flask.

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 continue stirring. The mixture should turn brown due to the presence of I_2 in the mixture.

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

Record the initial burette reading with two decimal places and then titrate the KIO_3 mixture (in flask) with the $Na_2S_2O_3$ solution until the color changes to *a very pale yellow*. *(no measurement needed at this point)*

Add approximately 3 mL of starch solution to the flask.

The reaction mixture should now be blue signifying the presence of the I_3- 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*. This is the endpoint and it is very sharp.

Record the final buret reading (2 decimals).

Repeat the standardization a second time using another 10 mL from your standard solution. Do not remake another standard solution.

All waste solutions may be flushed down the sink.

Determination of Sodium Hypochlorite in Bleach

Mix approximately 1 gram of solid KI with 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 (~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.

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

Refill the burette with $Na_2S_2O_3$ as needed.

Gently stir the mixture in the flask with a magnetic stir bar and titrate the contents with the $Na_2S_2O_3$ solution just as you did for the standardization trials.

Add 3 mL of starch once the solution has turned from brown to pale yellow.

Hand swirl the mixture to rinse any solution adhering to the sides of the flask.

Continue titrating slowly until the blue color disappears and record the burette measurement with 2 decimal places.

Repeat the experiment a second time using a new sample of the **same bleach sample** previously used.

V. Data Table: Standardization of $Na_2S_2O_3$

Use <u>excess significant figures</u> at all times unless asked to round.

Molar mass KIO_3 = 214.000984 g/mol

	Trial 1	Trial 2
mass of KIO_3 (g)		
total moles of KIO_3		
concentration KIO_3 solution (M)		
volume KIO_3 titrated(mL)		
A moles of KIO_3 titrated		
Initial Burette Reading (mL)		
Final Burette Reading (mL)		
Volume $Na_2S_2O_3$ dispensed (mL)		
moles $Na_2S_2O_3$ (calculated from *A*)		
Concentration $Na_2S_2O_3$ (M)		
*B*Average $Na_2S_2O_3$ concentration		

VI. Data Table: Titration of Bleach

Use excess significant figures at all times unless asked to round.

Molar mass NaClO = 74.441713 g/mol

OSHA regulations require we label the $Na_2S_2O_3$ solution as 0.05 Molar.
Under no circumstances should you use 0.05 M in any calculations.

	Trial 1	Trial 2
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 *B*)		
***C* moles $Na_2S_2O_3$**		
Moles ClO^- titrated (Use *C*)		
Mass NaClO (g) in sample		
Mass % NaClO		
Average Mass % *(Round only here with correct significant figures)*		

mass % $_{NaClO}$ = mass $_{NaClO}$ / mass $_{bleach}$ x 100 %