

Redox Titrations

Introduction

In this experiment you are going to use a pair of oxidation-reduction reactions to determine the concentration of a bleach unknown. First, you are going to standardize a freshly prepared solution of sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$). You will do this by reacting iodate (IO_3^-) with excess iodide (I^-) in acidic solution to produce a known quantity of iodine and water. The iodine produced will be titrated with a sodium thiosulfate solution. The products of the reaction between iodine and thiosulfate are the iodide ion and the tetrathionate ion ($\text{S}_4\text{O}_6^{2-}$). By using a known amount of iodate and stoichiometric calculations you are able to calculate the exact concentration of the thiosulfate solution you have prepared.

In the second part of the experiment you will use your standardized thiosulfate solution to analyze a bleach unknown. Bleach contains sodium hypochlorite (NaClO) that reacts with iodide to produce iodine, chloride ion, and water. The amount of iodine produced can then be determined by titration with your standard sodium thiosulfate.

As prelaboratory preparation, you should **read sections 5-4 to 5-6 in your Petrucci textbook, 8th ed.** Redox reactions are much more complicated than simple acid/base reactions. You should balance all the reactions described in this experiment before coming to the laboratory.

Safety: Wear gloves when handling bleach. Wear your goggles.

Procedure

Work individually on this experiment.

Part I Standardization of Sodium thiosulfate solution

Plan out a titration procedure for the standardization of a sodium thiosulfate solution. The following chemicals will be provided for you in the lab:

Solid potassium iodate (Must be dried in oven)
Solid potassium iodide
3 M sulfuric acid
1 M sodium thiosulfate
3% ammonium molybdate solution (This is a catalyst.)
Starch solution (This is the indicator you will use)
Bleach unknown

You should dilute the sodium thiosulfate solution to approximately 0.05 M. This will give you reasonable volumes for your titrations. Plan on making about 300 mL of the sodium thiosulfate solution.

Do enough standardization titrations (at least 3 trials) so that you can be sure of your results, i.e., can you perform a meaningful statistical analysis of your results?

Do not contaminate the chemicals. The solutions in the reagent bottles should be colorless. If they are brown they have been contaminated and should be returned to the dispensary. The most common source of contamination is due to leaving caps off the chemical bottles and using the same disposable pipet for more than one chemical. Be vigilant! Do not be the person in your lab room that causes these problems!

Below is a rough outline of the titration procedure; you should elaborate and provide further details such as the amounts used of each reagent in your laboratory report. The outline of the procedure also contains some important information that will assist you in performing an effective titration.

1. Preparation of the iodine solution.
 - Prepare 250 mL solution of potassium iodate, and transfer a measured amount of this solution into the titration flasks. This will speed up your titrations in two ways. You will spend far less time at the balance, and potassium iodate takes a few minutes to dissolve so you only have to wait once instead of several times.

It is recommended that you make a solution that is approximately 0.01 M. The solution does not have to be exactly 0.01 M, but you will need to calculate the exact concentration of the solution you prepare. You can prepare this in a 250 mL volumetric flask and share with **one other lab mate**. Be sure to use volumetric glassware to transfer this iodate solution into your titration flask

- In acidic solution Iodate and Iodide react to produce iodine. Potassium iodide and 3 M Sulfuric Acid has been provided. Sulfuric acid is chosen for this reaction since the sulfate anion will not interfere with this particular set of redox reactions. This mixture will turn brown.

- Since you know very precise information about your iodate solution, make iodate your limiting reagent. If you do your reactions with about **20 mL of iodate** solution you will need about two grams of potassium iodide and a few milliliters of the 3 M sulfuric acid. **Use your 10.00 mL transfer pipet (twice) to measure 20.00 mL the Potassium Iodate.**

- Save time by measuring only your limiting reagent carefully. The excess reagents can be measured out much more generally. On the other hand, do not waste chemicals by adding large excesses of the other reagents.

- Add enough deionized water to the titration flask so that you can easily see color changes.

2. Titration of the iodine solution with the sodium thiosulfate.

- Titrate the iodine solution with the sodium thiosulfate solution you have made. Do not add the starch indicator until the titration is almost finished. In concentrated iodine solutions the starch complex can tie up some of the dissolved iodine molecules, effectively removing them from the titration and affecting the accuracy of the results. As you perform the titration the solution should go from brown to yellow. Once the solution fades to a light yellow you should add the starch indicator. This sharpens the endpoint.

- Perform the titration immediately upon mixing the chemicals. Though it is tempting to use "assembly line" techniques and prepare all of your flasks at once and then titrate them all, this can also adversely affect your results. The iodine in the solution can clump together over time and become more and more difficult to dissolve.

Report your average Molarity of $\text{Na}_2\text{S}_2\text{O}_3$ standardized solution, and standard deviation. The post-lab exercises will guide you through these calculations.

Part II Analysis of your bleach unknown

In order for the on-line program to identify the unknown that you were assigned to analyze, you will need to know the hyphenated number embossed on your locker in your laboratory room. For example, in room 0435 Chemistry Annex one of the locker's hyphenated number reads, 0435-6-24; in room 66 Chemistry one of the locker's hyphenated number reads, 66-4-1.

Your locker's hyphenated number is _____ - _____ - _____.

Once you have standardized your sodium thiosulfate solution you can move on to the analysis of your bleach unknown. Plan a titration procedure that will allow you to determine the mass percentage of sodium hypochlorite in a bleach sample. Your procedure should be fundamentally the same as for the standardization, but hypochlorite will take the place of iodate in the reactions. **Plan to use a little less than a gram of bleach unknown in each of your titrations.** Record the mass of bleach used for each trial to a thousandth of a gram.

The tips for the standardization procedure also apply for your unknown analysis. Additionally, you will want to add about three milliliters of the ammonium molybdate catalyst solution to the hypochlorite-iodide reaction in order to speed it up. If you use single gram samples of bleach you will be able to use the same amounts of the other reagents that you used in the standardization.

Report the strength of the bleach as the effective mass percentage of the bleach that is sodium hypochlorite, NaClO , for each titration and then report the average mass percentage of sodium hypochlorite in the unknown bleach. Calculate a standard deviation and a 90% confidence limit. Post-laboratory exercise will guide you through each of the above calculations and questions.

<p>Clean up. Rinse the buret and all glassware with deionized water. All solutions can be disposed of down the drain. If time permits, now would be a good time to clean any other dirty glassware in your locker. Be sure that all glassware is returned to the proper place and that your laboratory bench has been rinsed with water using a sponge.</p>
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Post-Laboratory Exercise Questions

The following series of questions pertains to Part I of the Redox Titration Experiment, where you are to calculate the molarity of the sodium thiosulfate standard solution.

Enter the precise mass in **GRAMS** of the potassium iodate used to prepare your primary standard solution. Your mass precision should be reported to a thousandth of a gram. (Use 3 significant figures.)

If one had weighed out precisely 0.500 g of KIO_3 for the primary standard solution and dissolved it in enough deionized water to make a 250 mL solution, the molarity of that solution would be 0.00935 M. Enter your calculated molarity of the primary standard KIO_3 solution. Use 3 significant figures

Standardization of the sodium thiosulfate solution using the potassium iodate primary standard solution. We must examine each of the three acceptable trials. First, let's consider the analyte volume. You were instructed to pipet two 10.00 mL aliquots of potassium iodate into an Erlenmeyer flask for each analyte sample to be titrated. Therefore, your analyte volumes of the potassium iodate should be 20.00 mL. For each trial, enter the precise volume in mL of potassium iodate solution used in the standardization of sodium thiosulfate (e.g. 20.00 mL or 10.00 mL).

Standardization of the sodium thiosulfate solution using the potassium iodate primary standard solution. Now, we will examine the volumes of titrant, sodium thiosulfate, used in each trial. Your titration volumes of the sodium thiosulfate solution should be approximately 20 - 30 mL. For each trial, enter the precise volume in mL of sodium thiosulfate solution used in the titration of your potassium iodate solution (e.g. 20.34 mL).

Using the volumes of sodium thiosulfate solution you just entered and the molarity of the potassium iodate primary standard solution, calculate the molarity of the sodium thiosulfate secondary standard solution for each trial. Enter your calculated molarity of the sodium thiosulfate solution for each of the trial. Be sure to enter your calculated molarities in the corresponding order that you entered your sodium thiosulfate volumes previously. The sodium thiosulfate volume you entered for entry #1 above should correspond to the sodium thiosulfate molarity that you enter for entry #1 here. Use 3 significant figures.

The molarity of the sodium thiosulfate solution is taken as the average of the three trials. Please enter the average molarity using 3 significant figures.

Please enter the standard deviation of the sodium thiosulfate molarity.

The following series of questions pertains to Part II of the Redox Titration Experiment, where you are to calculate the mass percent of sodium hypochlorite in your sample of bleach.

For the reaction of hypochlorite anion with iodide anion, the iodide anion acts as the reducing agent according to the oxidation half-reaction, $2 \text{I}(\text{aq}) \rightarrow \text{I}_2(\text{aq}) + 2\text{e}^-$. Which of the following reduction half-reactions is correct to give the overall reaction of hypochlorite anion with iodide anion? 3 choices will be given.

For the reaction of thiosulfate anion with iodine, the thiosulfate anion acts as the oxidizing agent according to the oxidation half-reaction, $2 \text{S}_2\text{O}_3^{2-}(\text{aq}) \rightarrow \text{S}_4\text{O}_6^{2-}(\text{aq}) + 2\text{e}^-$. Which of the following reduction half-reactions is correct to give the overall reaction of thiosulfate anion with iodine? 3 choices will be given.

Notice that the effective analytical connection for analysis of hypochlorite anion by the thiosulfate anion in this experiment is the sum of the two reactions: 1) hypochlorite anion with iodide anion to form iodine, and 2) thiosulfate anion with the iodine formed in reaction one. In your laboratory notebook sum these two reactions to find the stoichiometric factor that relates moles of thiosulfate anion needed to react with each mole of hypochlorite anion in the bleach sample. Then select the correct response. 3 choices will be given.

For each of 3 acceptable trials, enter the mass of your bleach sample in **GRAMS**. Your mass precision should be reported to a thousandth of a gram.

For each of the 3 acceptable trials, enter the precise volume in **milliliters** of sodium thiosulfate solution used in the titration of your bleach samples (e.g. 20.34 mL). Be sure to enter your trial volumes in the corresponding order that you entered your masses of your bleach samples previously. The bleach sample mass you entered for entry #1 above should correspond to the sodium thiosulfate volume you enter for entry #1 here.

Using the volumes of sodium thiosulfate solution you just entered, the mass of bleach sample, and the average molarity of the sodium thiosulfate solution entered earlier, calculate the mass percent of NaClO for each bleach sample. Enter the calculated mass percent of NaClO in each of the 3 acceptable trials. Be sure to enter your mass percent values in the corresponding order that you entered your masses of your bleach samples and volumes of your sodium thiosulfate previously. The bleach sample mass you entered for entry #1 and the volume of sodium thiosulfate you entered above should correspond to the mass percent of NaClO you enter for entry #1 here. Use 3 significant figures.

Using your 3 trial values calculate the average mass percent of NaClO. Enter the average mass percent of NaClO in the bleach sample. (Use 3 significant figures)

Please enter the standard deviation of the average mass percent of NaClO in your bleach sample. Please enter the calculated 90% confidence limit for the average mass percent of the NaClO in your bleach samples.

We will now determine the percent error in your analysis of the percent mass of NaClO in your bleach sample. In order for us to identify which unknown sample you analyzed, you will need to enter your laboratory room number, locker series and locker number. These numbers are the hyphenated numbers embossed on your locker. For example, if the hyphenated numbers read, 0435-6-24, your room number is 0435, your locker series is 6, and your locker number is 24. If the hyphenated numbers are 66-4-1, your room number is 66, your locker series is 4, and your locker number is 1.

Concluding Remarks: Briefly discuss interpretations of your observations and results. Include in your discussion, any conclusions drawn from the results and any sources of error in the experiment.