Chemistry 120: Experiment 3

Preparation of Standard Sodium Thiosulfate Solution and Determination of Hypochlorite in a Commercial Bleach Product

Iodine can be used as an oxidizing agent in many oxidation-reduction titrations and iodide can be used as a reducing agent in other oxidation-reduction titrations:

$$I_2 + 2\,e^- = 2\,I^- \qquad (1)$$

If a standard iodine solution is used as a titrant for an oxidizable analyte, the technique is iodimetry. If an excess of iodide is used to quantitatively reduce a chemical species while simultaneously forming iodine, and if the iodine is subsequently titrated with thiosulfate, the technique is iodometry. Iodometry is an example of an indirect determination since a product of a preliminary reaction is titrated.

The use of iodine as a titrant suffers from two major disadvantages. First, iodine is not particularly soluble in water, and second, iodine is somewhat volatile. Consequently, there is an escape of significant amounts of dissolved iodine from the solution. Both of these disadvantages are overcome by adding iodide (I^-) to iodine (I_2) solutions. In the presence of iodide, iodine reacts to form triiodide (I_3^-) which is highly soluble and not volatile.

$$I_2 + I^- = I_3^- \qquad (2)$$

The major chemical species present in these solutions is triiodide. The reduction of triiodide to iodide is analogous to the reduction of iodine.

$$I_3^- + 2\,e^- = 3\,I^- \qquad (3)$$

Triiodide reacts with thiosulfate to yield iodide and tetrathionate.

$$\underset{\text{thiosulfate}}{2\,S_2O_3^{2-}} + I_3^- = \underset{\text{tetrathionate}}{S_4O_6^{2-}} + 3\,I^- \qquad (4)$$

Dilute triiodide solutions are yellow, more concentrated solutions are brown, and even more concentrated solutions are violet. Iodide solutions are colorless. If all of the other solution components are colorless, it is possible to detect the endpoint of titrations involving triiodide without the use of an indicator. Endpoint detection is considerably easier, however, with an indicator. The indicator that is usually chosen for titrations involving iodine (triiodide) is starch. Starch forms a dark blue complex with iodine. The end point in iodimetry corresponds to a sudden color change to blue. Likewise the end point in iodometry corresponds to a sudden loss of blue color due to the complex. Potato starch, rather than corn starch, is preferred for making the indicator solution since the color change due to the starch complex at the end point is sharper. In iodometry the starch is added only after the color due to triiodide has begun to fade, i.e., near the endpoint, because starch can be destroyed in the presence of excess triiodide.

In the first portion of this experiment a sodium thiosulfate solution is prepared and standardized with the primary standard potassium iodate. Iodate (IO_3^-) reacts with an excess of

iodide in acid solution to yield triiodide, which is subsequently titrated with the standardized thiosulfate solution.

$$IO_3^- + 8\,I^- + 6\,H^+ = 3\,I_3^- + 3\,H_2O \qquad (5)$$

The standardization is an example of iodometry. The standardized thiosulfate is used in the second portion of the experiment to determine the hypochlorite ion in bleach as discussed below.

Apparatus

1.5- or 2-L beakers
1-L storage bottle
250-mL Erlenmeyer flasks, three
10-mL graduated cylinder
2-mL pipette
50-mL burette
burners and tripods

Chemicals

hydrochloric acid (6 M)
potassium iodate
potassium iodide
sodium carbonate
sodium thiosulfate pentahydrate
soluble potato starch
sulfuric acid (3 M)
Chlorox or similar liquid bleach as an unknown

Preparation of Standard Sodium Thiosulfate Solution

1. Dissolve approximately 0.1 g of sodium carbonate in one liter of distilled water. The sodium carbonate is added to adjust the pH of the solution to about 9 or 10. Dry the primary standard KIO_3 at 110 °C for 1 hr or confirm that it has already been dried.

2. Prepare a 0.1-M thiosulfate solution by dissolving approximately 25 g of sodium thiosulfate pentahydrate ($Na_2S_2O_3 \cdot 5H_2O$; FW 248.19) in the sodium carbonate solution. Transfer the solution to a clean storage bottle, and store the solution in the dark.

3. The next three steps involve preparation of the indicator. Enough indicator for an entire 20-student section can be prepared as follows. (Your laboratory instructor will designate three people to prepare the community supply of indicator). Prepare a paste by adding about 2 g of soluble starch to 30 mL of deionized water. The starch serves as the indicator for titrations involving iodine.

4. Boil about 1 liter of water in a 1.5- or 2-L beaker and add the paste. Stir the solution while continuing to heat until the solution is completely transparent. If the solution remains cloudy after at least 15 min of heating, continue to step 5. The solution is still usable.

5. Cool the starch indicator solution and place it in a clean storage bottle (LABEL IT!).

6. Fill a 50-mL burette with the thiosulfate solution.

7. Weigh to ± 0.1 g approximately 2 g of reagent-grade potassium iodide into each of three numbered 250-mL Erlenmeyer flasks. Add 25 mL of water and 2 mL of 6 M hydrochloric acid to each flask. Swirl the solution to dissolve the potassium iodide.

8. Number three weighing bottles or three small beakers with the same numbers used for the Erlenmeyer flasks.

9. Into each weighing bottle or beaker weigh to the nearest 0.1 mg between 0.12 and 0.15 g of potassium iodate (FW 214.00). Record the mass of the potassium iodate in each container.

10. Add the potassium iodate in <u>one</u> of the numbered containers to the solution in the identically numbered Erlenmeyer flask. Rinse in any remaining particles with a small amount of water. Swirl the solution to dissolve the potassium iodate. The brown color of triiodide should be apparent in the mixed solution.

11. Titrate the solution in the Erlenmeyer flask with the thiosulfate solution until the triiodide color has become noticeably less intense. Add 5 mL of the starch indicator and continue the titration until the dark-blue color of the starch-triiodide complex just disappears. Record the endpoint volume to the nearest 0.01 mL, and use fractional drops if necessary.

12. <u>Individually</u> repeat steps 10 and 11 with the remaining two potassium iodate samples. To minimize error, generate the triiodide just before you titrate it with thiosulfate.

Calculations - Preparation of Standard Sodium Thiosulfate Solution

1. From the mass of the potassium iodate and the endpoint volume for each of the titrations, calculate three values of the concentration of the thiosulfate solution. You will need the stoichiometric relationships embodied in equations 4 and 5 above.

2. Determine the mean thiosulfate concentration (in units of molarity), the standard deviation and the relative standard deviation of your results.

Iodometric Determination of Hypochlorite in Commercial Bleach Product

In most liquid laundry bleaches and in some solid bleaches, the active ingredient is hypochlorite (OCl^-). Liquid bleaches usually contain sodium hypochlorite and the bleaching action is caused by the strong oxidizing properties of OCl^-, which also are exploited in this iodometric determination of hypochlorite. In the experiment, iodide is used to reduce the hypochlorite in bleach. The reaction in acid solution yields chloride and triiodide.

$$OCl^- + 3\,I^- + 2\,H^+ = Cl^- + I_3^- + H_2O \qquad (6)$$

The triiodide formed in this reaction is titrated with the standard thiosulfate solution that was prepared earlier.

$$2\,S_2O_3^{2-} + I_3^- = S_4O_6^{2-} + 3\,I^- \qquad (4)$$

thiosulfate ↑ ↑ tetrathionate

The endpoint of the titration is located with the starch indicator solution that was also prepared earlier.

1. Add 50 mL of deionized water and between 1.5 and 2.0 g of potassium iodide to each of three 250-mL Erlenmeyer flasks. Stir the solutions until the potassium iodide in each flask is dissolved.

2. Add 10 mL of 3-M sulfuric acid to each flask.

3. Use a pipette to place exactly 2.00 mL of liquid bleach into one of the flasks, and stir by swirling the solution. Fill a 50-mL burette with standard sodium thiosulfate solution, and use the thiosulfate solution to titrate the triiodide formed during the reaction of bleach with iodide. When the triiodide color starts to fade, add 5 mL of starch solution and continue the titration to the endpoint. Record the endpoint volume to the nearest 0.01 mL.

   CAUTION: Liquid bleach is a mixture of sodium hypochlorite and sodium hydroxide. It is very corrosive. Do not allow the bleach to come in contact with your skin.

4. Individually repeat step 3 with the solutions in each of the other two Erlenmeyer flasks. Again, generate iodine just before the titration with thiosulfate.

Calculations - Iodometric Determination of Hypochlorite

1. Use the volume of sample (2.00 mL), the endpoint volume, and the mean concentration of the thiosulfate solution to determine values for the concentration (in units of molarity) of hypochlorite in the bleach product for each of the three titrations. Report these values, and their mean, standard deviation, and relative standard deviation.

Report. Using the format specified, prepare a lab report summarizing the data for your thiosulfate standardizations (masses of KIO_3, volumes of thiosulfate solution, mean thiosulfate concentration and standard deviation). Also include results for the determination of hypochlorite in bleach (mean molarity and its standard deviation).

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