EXPERIMENT 21B

Quantitative Redox Reactions Involving Iodine

Redox reactions are involved in a wide variety of techniques for quantitative analysis of chemical substances. The methods for calculating the amounts of substances involved in these reactions (expressed either as mass in grams or as the volume of a solution of known molarity) were introduced in Chapter 6 of the *Heath Chemistry* text. The balanced equations for many of these reactions are complex, and must be obtained either by adding half-reaction equations or by using the oxidation number method, as outlined in Chapter 21 of the *Heath Chemistry* text.

A substance that is often used in quantitative redox reactions is iodine, I_2. It is easily formed by oxidizing I^-, but the reaction can be reversed just as easily, so a wide variety of chemicals can be reacted with iodine. In addition, it gives a characteristic deep blue color with starch solution even when it is in very low concentrations, making it easy to determine when the iodine is all used up, or when it begins to form. Starch is therefore used as the indicator in these reactions.

In Part I of this experiment you will prepare a solution of potassium iodate (KIO_3) of known concentration. This solution makes a good primary standard in redox titrations, since it is stable and can be obtained very pure. In Part II, known volumes of this solution will have excess H^+ and I^- added, and a reaction will occur in which I_2 is produced in a quantity determined by the moles of KIO_3 present initially. A solution of sodium thiosulfate, $Na_2S_2O_3$, of unknown concentration will then be titrated into the solution, which will react with the I_2. Starch will be added when most of the I_2 has reacted with $S_2O_3^{2-}$, and the titration will be continued until the blue color disappears. The concentration of the $Na_2S_2O_3$ solution can now be calculated. This solution can be used to determine the amount of I_2 in other solutions. The further analyses involved in this experiment are to determine the $[Cu^{2+}]$ in a solution (by adding I^-, which reduces Cu^{2+} to copper(I) iodide, CuI, and becomes I_2 in the process) (Part III) and to determine the amount of vitamin C in a sample by analyzing it with a standard I_2 solution (Part IV).

OBJECTIVES

1. to prepare a solution of potassium iodate, KIO_3, of known molarity
2. to standardize a solution of sodium thiosulfate, $Na_2S_2O_3$, by titrating it against standard KIO_3 (with excess I^-, H^+)
3. to determine the molarity of Cu^{2+} in an unknown sample by reacting it with I^- and titrating the liberated I_2 with standard $S_2O_3^{2-}$
4. to use a standard I_2 solution to determine the mass of ascorbic acid (vitamin C) in a juice sample or a vitamin tablet

MATERIALS

Apparatus

centigram balance
3 beakers (100 mL)
funnel
wash bottle
volumetric flask (250 mL)
buret (50 mL) pipet (25 mL)
Erlenmeyer flask (250 mL)
graduated cylinder (10 mL)
beaker (250 mL)
bottle (500 mL) with stopper
graduated cylinder (100 mL)
lab apron
safety goggles
suction bulb

Reagents

solid potassium iodate, KIO_3
1*M* potassium iodide, KI
1*M* sulfuric acid, H_2SO_4
sodium thiosulfate solution, $Na_2S_2O_3$ (approx. 0.12*M*)
starch solution
copper(II) sulfate solution, $CuSO_4$ (unknown concentration)
iodine in potassium iodide solution
250 mg or 300 mg vitamin C tablets
orange or other citrus juice

PROCEDURE

Part I Preparation of a Standard Potassium Iodate Solution

1. Before coming to the laboratory, calculate the mass of KIO_3 required to make up 250 mL of a 0.0200*M* solution of KIO_3.
2. Put on your lab apron and safety goggles.
3. Accurately measure the mass of a clean, dry 100 mL beaker and record it in your copy of Table 1 in your notebook.
4. Place solid KIO_3 in the beaker until you have approximately the mass calculated in Step 1. Record the actual mass accurately in Table 1.
5. Dissolve the KIO_3 in water, and pour the solution through a funnel into a 250 mL volumetric flask. Rinse the beaker twice, and add the rinsings to the flask.
6. Add water until the level of the solution is up to the mark. Stopper the flask, and shake it thoroughly to make the solution homogeneous.

CAUTION: Potassium iodate solution is poisonous. Do not get any in your mouth, and do not swallow any. Always use a suction bulb on the pipet.

Part II Standardization of Sodium Thiosulfate Solution

1. Transfer the standard KIO_3 solution to a clean, dry 250 mL beaker.
2. Obtain in 100 mL beakers approximately 50 mL each of 1*M* KI, 1*M* H_2SO_4, and starch solution. Label each beaker.
3. Using a suction bulb on your pipet, withdraw 25 mL of the KIO_3 solution and transfer it to a 250 mL Erlenmeyer flask, touching the tip to the side of the flask to ensure that the correct volume is delivered.
4. Add to the flask containing the KIO_3 solution approximately 5 mL of 1*M* KI and 5 mL of 1*M* H_2SO_4. These are excess amounts, so precise measurement is not required. Use a 10 mL graduated cylinder for adding the KI and H_2SO_4. (You will notice that a brown precipitate is formed at first, and that it dissolves again to give a clear brown

CAUTION: Sulfuric acid is very corrosive. Wash any spills and splashes on skin or clothing with plenty of water. Call your teacher.

solution. This behavior is characteristic of iodine, I_2. The amount of precipitate formed is determined by the moles of KIO_3 originally present. The iodine will now be made to react with sodium thiosulfate solution, $Na_2S_2O_3$, in order to determine the molarity of the $Na_2S_2O_3$.)

5. Fill a 500 mL bottle with the approximately 0.12*M* $Na_2S_2O_3$ solution provided. Add about 15 mL of the solution to the buret, rinse, and discard.
6. Fill the buret with $Na_2S_2O_3$, then open the valve to allow some to drain through the tip.
7. Close the valve. The tip should be filled with the solution. Read the initial volume of $Na_2S_2O_3$ in the buret and record it in Table 2.
8. Run the $Na_2S_2O_3$ solution into the flask, swirling constantly, until the brown color has faded to a light yellow. Add 5 mL of starch solution, and continue adding the $Na_2S_2O_3$ drop by drop until the blue-black color disappears. Note that the endpoint is a very precise one, and that it is therefore important not to add the starch too soon. Read the final volume in the buret and record it in Table 2.
9. Repeat Steps 3 to 8 until you obtain consistent results. You now have enough data to calculate the molarity of the $Na_2S_2O_3$ solution.

Part III Determination of the Concentration of an Unknown Solution of Copper(II) Sulfate

CAUTION: Copper(II) sulfate solution is poisonous. Do not get any in your mouth. Do not swallow any.

1. Obtain in a beaker approximately 100 mL of the unknown $CuSO_4$ solution provided. Write down its identifying letter or number if more than one unknown is provided.
2. Using a suction bulb, withdraw into a pipet 25 mL of the $CuSO_4$ solution and transfer it to a 250 mL Erlenmeyer flask. Remember—rinse the pipet with the solution first.
3. Add 10 mL of 1.0*M* KI solution to the $CuSO_4$. Use a graduated cylinder; a precise volume is not required.
4. The cloudy brown material produced in the flask consists of brown I_2 solution and a precipitate of white copper(I) iodide, CuI. The iodine can now be titrated with $Na_2S_2O_3$. The CuI does not interfere with this reaction.
5. Read the initial volume of $Na_2S_2O_3$ in the buret and record it in Table 3. Run the solution into the flask until the brown color has faded to light yellow. Add 5 mL of starch solution, and continue adding $Na_2S_2O_3$ drop by drop until the blue-black color disappears. Read and record the volume in the buret. (A light-colored precipitate of CuI will remain.)
6. Repeat Steps 2 to 5 once or twice more until consistent results are obtained. You now have enough data to calculate the molarity of Cu^{2+}, and, therefore, the molarity of the $CuSO_4$ solution.

Part IV Determination of Amount of Vitamin C in a Sample

1. Obtain in a 250 mL beaker about 200 mL of the solution of iodine (in potassium iodide) provided. You will need to standardize it before doing the rest of this part.

2. Using a suction bulb, withdraw 25 mL of the iodine solution into a pipet and transfer it to a 250 mL Erlenmeyer flask.
3. Refill the buret with the standardized $Na_2S_2O_3$ solution, take the initial reading, and record it in Table 4.
4. Run the $Na_2S_2O_3$ solution into the flask until the brown color of the I_2 fades to pale yellow, add 5 mL starch solution, and continue the titration dropwise until the dark blue color just disappears. Again read the final volume in the buret and record it in Table 4.
5. Repeat Steps 2 to 4 until consistent results are obtained. You now have enough data to calculate the molarity of the I_2 solution.
6. Discard the $Na_2S_2O_3$ solution remaining in the buret, wash the buret with water, then rinse it with the I_2 solution. Discard this, then fill the buret again with I_2, and drain some to refill the tip.
7. Obtain a 250 mg (or 300 mg) tablet of vitamin C (ascorbic acid) and place it in an Erlenmeyer flask. Add about 50 mL of water, and shake to dissolve. Add 5 mL of starch solution.
8. Read the initial volume of the I_2 solution in the buret and record it in Table 5. Then allow it to run into the flask. Swirl constantly, watching for the first appearance of the characteristic dark blue color which stays even after swirling. Again read the final volume of the I_2 solution in the buret and record it in Table 5.
9. Repeat Steps 7 and 8 until consistent results are obtained.
10. Obtain a sample of orange or other citrus juice (or other fruit juice to which vitamin C has been added). Using a graduated cylinder, measure out 100 mL of the juice and pour it into a 250 mL Erlenmeyer flask. Add 5 mL of starch solution.
11. Read the initial volume of I_2 solution in the buret and record it in Table 6. Then run it into the flask, swirling until the first permanent blue-black color is produced. Again read the final volume of I_2 solution in the buret and record it in Table 6.
12. Repeat, using another sample or another juice if requested to do so by your teacher.
13. Before leaving the laboratory, wash your hands thoroughly with soap and water; use a fingernail brush to clean under your fingernails.

CAUTION: Iodine causes burns and is a strong irritant to eyes and skin. If any is spilled on your skin, wash first with sodium thiosulfate solution, then with plenty of water.

REAGENT DISPOSAL

Rinse solutions that are left over in the flask after the titrations down the sink with copious amounts of water. Return any leftover KIO_3, KI, $Na_2S_2O_3$, $CuSO_4$, and I_2 in KI solution to the containers designated by your teacher.

POST LAB DISCUSSION

In order to calculate the results for this experiment, you will need to work out the balanced overall redox equation for each reaction that is occurring, then use the mole relationships in the equation to relate reacting quantities.