The Iodine Clock Reaction

It is very important for a chemist to understand the conditions that affect the rate of a chemical reaction. In chemical manufacturing processes, controlling the rate of a given reaction can make all the difference between an economical process and an uneconomical one.

In the previous experiment (11A) you investigated the principle that the rate of a reaction is determined by several factors, namely: concentration of reactants, temperature, surface area of reactants (for a heterogeneous reaction), nature of reactants, and the presence of a catalyst. In this experiment, you will carry out a different reaction, but you will again investigate the effect of the concentration of reactants (in Part I) and the temperature (in Part II) on the reaction rate.

This experiment involves a reaction that is sometimes called an iodine clock reaction. There are a number of different combinations of chemicals that give a reaction of this type. What happens, essentially, is that there are two different reactions: one in which iodine is produced (a slow reaction) and one in which the iodine produced in the first reaction is used up (a fast reaction). By carefully controlling the quantities of reactants, you can obtain a situation in which the reactant in the second reaction is used up first, allowing iodine to form at that point. At very low concentrations the iodine then combines with starch to suddenly give a deep blue-black color, at a time determined by the conditions used. Hence the term “iodine clock”. The time elapsed from when the solutions were first mixed together until the point when the blue-black color appears is measured, and from this time measurement the rate of the reaction can be determined. You will alter the conditions of concentrations of reactants in Part I and temperature in Part II in order to determine their effect on reaction rate.

OBJECTIVES

1. to observe and record the effect of changing the concentration of a reactant on the rate of a reaction
2. to observe and record the effect of changing the temperature of a system on the rate of a reaction
SUPPLIES

Equipment

test-tube rack 4 beakers (250 ml.)
2 beakers (100 ml.) electric kettle or hot plate
2 graduated cylinders (10 ml.) lab apron
8 test tubes (18 mm x 150 mm) safety goggles
2 dropping pipet stopwatch or other timing device
Thermometer ice

Chemical Reagents
Solution A: 0.020M KIO₃ (potassium iodate)
Solution B: 0.0020M NaHSO₃ (sodium bisulfite)
(also containing 4 g of starch and 12 mL of 1M H₂SO₄/L)

PROCEDURE

Part 1: Effect of Concentration

1. Put on your lab apron and safety goggles
2. Obtain in separate 100 mL beakers about 60 mL of Solution A (0.020M KIO₃) and 90 mL of Solution B (0.0020M NaHSO₃ containing H₂SO₄ and starch) and label the beakers.
3. In a 10 mL graduated cylinder place 10.0 mL of Solution A, using a dropping pipet, to obtain the volume as accurately as possible. Transfer the solution to an 18 mm x 150 mm test tube in a rack.
4. In the same manner measure out 10 mL of Solution B and transfer it to another test tube. Use a different graduated cylinder and dropping pipet than for Solution A and keep the same ones for each solution for subsequent parts of the procedure.
5. In order to measure the time needed for the reaction to occur you will need a watch or clock with a sweep second hand, or preferably a digital watch with a stopwatch function. One partner must record the time, while the other partner mixes the solutions. Mix the solutions in one of the two test tubes and record the time from the instant they first mix.
6. Very quickly pour the solution back and forth between the two test tubes three times to make sure they are thoroughly mixed, then wait for the completion of the reaction.
7. Record the time at the instant the deep blue-black color first appears.
8. In order to study the effect of changing the concentration of Solution A, half the class will be assigned the values of 9.0 mL, 7.0 mL, 5.0 mL, and 3.0 mL, and the other half the values of 8.0 mL, 6.0 mL, 4.0 mL, and 2.0 mL. In each instance, measure out the volume in the graduated cylinder and add enough water to make it up to the 10.0 mL mark. Then transfer each dilution of Solution A to a test tube and mix it with 10.0 mL Solution B as before. For each, record the time taken for the color to appear in your copy of Table 1 in your notebook.
9. Record your results on the board to get class averages.

**Part II: Effect of Temperature**

1. In this part of the procedure you will keep the concentration constant and vary the temperature, both above and below room temperature. In order that the reaction times lie in a suitable range, Solution A will be at only half the concentration of the original solution.

2. Make up 2 sets of each of solutions A and B, with 5.0 mL of Solution A and 5.0 mL of water in one set of four test tubes and 10.0 mL of Solution B in the other 4 test tubes.

3. Make 4 water baths (in 250 mL beakers) at temperatures 5°C, 15°C, 25°C, and 35°C, or 10°C, 20°C, 30°C, and 40°C, depending on which set of temperatures your instructor assigns you. Use ice to obtain the temperatures below room temperature and hot water from a tap, kettle, or beaker on a hot plate to obtain the temperatures above room temperature. The beakers should be about two-thirds full, so that the solutions in the test tubes are well beneath the level of the water in the baths.

4. Place one test tube containing diluted Solution A and another containing Solution B in each water bath and leave them for 10 min to allow them to adjust to the temperature required.

5. Try to maintain the temperatures in the water baths within 0.5°C of the temperatures assigned by adding ice or hot water as required. This will make comparisons with other groups in the class more meaningful.

6. When the temperatures are at the correct value and the tubes have been in for enough time, mix each pair of solutions into one test tube and then pour back and forth three times. Then place the test tube back in the water bath. In Table 2, record the time elapsed from the instant the solutions are mixed to the instant the blue-black color first appears. Do this for each pair of solutions at each temperature.

7. Record your results on the board in order to get class averages.

8. Before leaving the laboratory, wash your hands thoroughly with soap and water

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**REAGENT DISPOSAL**

All solutions remaining after each reaction may be safely rinsed down the sink with plenty of water. Your instructor will tell you what to do with the left over original solutions.

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**POST LAB CONSIDERATIONS**

The blue-black color observed in Parts I and II occurs as a result of two separate reactions. Initially, the iodate ion, IO$_3^-$, reacts with the bisulfate ion, HSO$_3^-$, giving iodide ion, I$^-$, and sulfate ion, SO$_4^{2-}$, as follows:

$$\text{IO}_3^-(aq) + 3\text{HSO}_3^-(aq) \rightarrow \text{I}^-(aq) + 3\text{SO}_4^{2-}(aq) + 3\text{H}^+(aq)$$

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**CAUTION!!!**

Your thermometer is made of glass and breaks easily, leaving sharp edges that cut. Handle your thermometer gently. If your thermometer breaks, call your instructor. If it contains mercury, be aware that mercury liquid and vapor are very poisonous.
The bisulfate ions are present in lower concentration and are therefore used up first. When this happens, the IO$_3^-$ ions then react with I$^-$ ions in the presence of H$^+$ ions to give molecular iodine, I$_2$:

$$\text{IO}_3^-(aq) + 5\text{I}^-(aq) + 6\text{H}^+(aq) \rightarrow 3\text{I}_2(aq) + 3\text{H}_2\text{O}(l)$$

In the presence of starch, iodine forms the intense blue-black color as a result of the iodine molecules being trapped in the long starch molecules. The appearance of this color indicates that the first reaction is complete and the second one has begun to take place.

The uncertainty in measuring the time taken for the reaction could easily be $\pm 2$ s unless you are very careful. Thus, the interpretation of results will be more meaningful if the class averages at a particular concentration are considered. Measuring the time taken for a reaction to be completed is not the same as measuring its rate, but there is an inverse relationship between them: rate is proportional to the reciprocal of time. Consequently, in interpreting the results you will calculate the rate in terms of reciprocal seconds ($s^{-1}$) and plot graphs of the rate against concentration or temperature.

### EXPERIMENTAL RESULTS

#### Part I: Effect of Concentration

**Table 1**

<table>
<thead>
<tr>
<th>Volume of KIO$_3$ (mL)</th>
<th>10.0</th>
<th>9.0</th>
<th>7.0</th>
<th>5.0</th>
<th>3.0</th>
</tr>
</thead>
<tbody>
<tr>
<td>Time for Reaction (s)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

(If you were assigned the other values of 8.0, 6.0, 4.0, and 2.0, use these instead.)

#### Part II: Effect of Temperature

**Table 2**

<table>
<thead>
<tr>
<th>Temperature ($^\circ$C)</th>
<th>5.0</th>
<th>15.0</th>
<th>25.0</th>
<th>35.0</th>
</tr>
</thead>
<tbody>
<tr>
<td>Time for Reaction (s)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

(If you were assigned the other values of 10.0$^\circ$C, 20.0$^\circ$C, 30.0$^\circ$C, 40.0$^\circ$C, use these instead.)