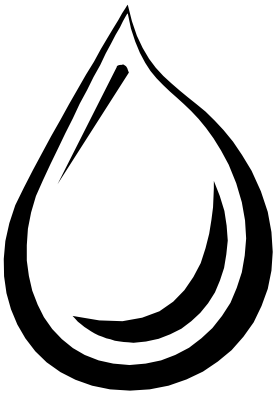
PREPARATION AND PROPERTIES OF ATMOSPHERIC GASES:
Part TWO: Sulfur Dioxide and Nitrogen Dioxide



Acid Rain

INTRODUCTION

Sulfur dioxide (SO_2) plays a large role in acid rain. The following reaction takes place in the earth's atmosphere:

$$2SO_2 + O_2 \text{ ----> } 2SO_3$$

The SO_3 generated then reacts with water vapor in the clouds to form sulfuric acid:

$$H_2O + SO_3 \text{ ----> } H_2SO_4$$

Nitric oxide (NO) and nitrogen dioxide (NO_2) also play a role in acid rain and are quite toxic. NO and NO_2 are directly linked to the internal combustion engines we use to fuel our cars and other large machinery.

In this experiment, you will prepare sulfur dioxide and nitrogen dioxide and then examine the nature of these two gases in terms of their aqueous solutions. SO_2 will be generated by reacting sulfuric acid (H_2SO_4) with sodium sulfite (Na_2SO_3) to form sodium sulfate (Na_2SO_4), water (H_2O), and sulfur dioxide gas (SO_2):

$$H_2SO_4 + Na_2SO_3 \text{ ----> } Na_2SO_4 + H_2O + SO_2$$

The NO_2 will be prepared in three steps. **Step 1** involves reacting H_2SO_4 with potassium nitrite (KNO_3) to form potassium sulfate (K_2SO_4), H_2O, potassium nitrate (KNO_3), and nitric oxide (NO):

Step 1 $H_2SO_4 + 3KNO_2 \text{ ----> } K_2SO_4 + H_2O + KNO_3 + 2NO$

In **Step 2**, oxygen will be generated by reacting hydrogen peroxide with iron (III) chloride as in Atmospheric Gases I:

$$\text{Step 2} \qquad 2H_2O_2\,(l) \xrightarrow[\text{catalyst}]{} 2H_2O\,(l) + O_2$$

With the NO generated in a large bag and the O_2 generated in a second, smaller bag, **Step 3** involves allowing the NO and O_2 to react to form nitrogen dioxide (NO_2):

$$\text{Step 3} \qquad 2NO + O_2 \longrightarrow 2NO_2$$

SAFETY AND DISPOSAL

- The liquids used in this experiment, 10% hydrogen peroxide (H_2O_2) and 6 M sulfuric acid (H_2SO_4), are corrosive materials and should be handled cautiously. Refer to page 6, section C number 3 and 4 regarding spills on yourself, clothing, or benchtop.
- Both NO_2 and SO_2 are toxic gases and should not be allowed to escape into the lab. GENERATE AND CONDUCT THE TESTS ON THESE GASES **ONLY** IN THE HOOD.
- Be sure to rinse all thin-stem, plastic pipets well with distilled water before using. Use labeled pipets only for the assigned chemical.

EXPERIMENTAL PROCEDURE

will appear to remind you of potential dangers and hazards.

I. Setting up the well plate

Your 24-well tray should still be set-up as described on Page 25.

II. Generating gases

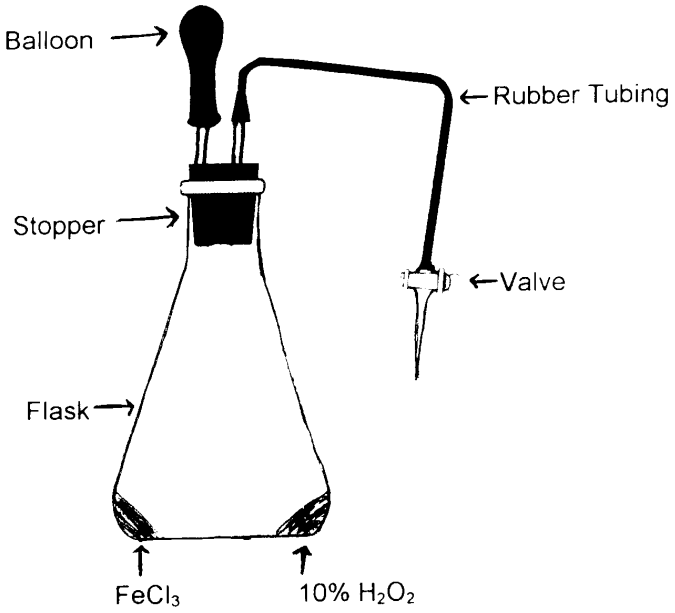
A. Sulfur dioxide

Work with this gas in the hood. Do not open the flask containing the gas outside the hood.

1. Place about 2 grams of sodium sulfite (Na_2SO_3) in the bottom to one side of the appropriately labeled flask.
2. Generously fill a thin-stem, plastic pipet with 6 M sulfuric acid (H_2SO_4). Don't put pipets into stock bottles of solutions. Dispense a small amount in a weigh boat or other available container.

6M sulfuric acid is a corrosive material. Be careful not to spill any on yourself, your clothing, or the benchtop

3. **Remember this is to be done in the hood. Check for proper placement of balloon on glass rod. Make sure valve is closed.** Place the 6M sulfuric acid (H_2SO_4) in the flask on the side opposite from the sodium sulfite (Na_2SO_3) and immediately restopper the flask. Mix the chemicals together by slowly shaking while holding stopper on flask.
4. KEEP THE FLASK CLOSED. The balloon located atop the stopper should start to expand. Record your observations.
5. You should now have a flask and balloon filled with SO_2.
6. Go to section III (Properties of carbon dioxide and oxygen) and perform all the tests dealing with SO_2.



B. Nitrogen dioxide

This gas will be generated in three steps. First, you will generate nitric oxide in a gallon Ziploc™ bag. Second, oxygen will be prepared in the same manner as in Atmospheric Gases I except you will use the quart-sized bag with the OneZip™ closure. Third, these two gases will be reacted to form nitrogen dioxide (NO_2).

NOTE: You will have to open the small bag containing oxygen inside the larger bag containing NO. As this is not an easy feat, you will probably want to practice the method a few times before proceeding just to make sure you can perform it successfully. The OneZip™ closure should make opening the small bag easier.

Work with this gas in the hood. **Do not** open the bag containing the gas outside the hood.

1. Preparation of the nitric oxide generating bag
   a. Weigh about 1.5 grams of potassium nitrite (KNO_2) and place it in the bottom corner of a 1-gallon, heavy-duty Ziploc™ bag.
   b. Fill a thin-stem, plastic pipet with 6 M H_2SO_4.

6M sulfuric acid is a corrosive material. Be careful not to spill any on yourself, your clothing, or the benchtop.

   c. Place the thin-stem, plastic pipet with 6 M H_2SO_4 in the plastic bag with the KNO_2.

2. Preparation of the oxygen generating bag

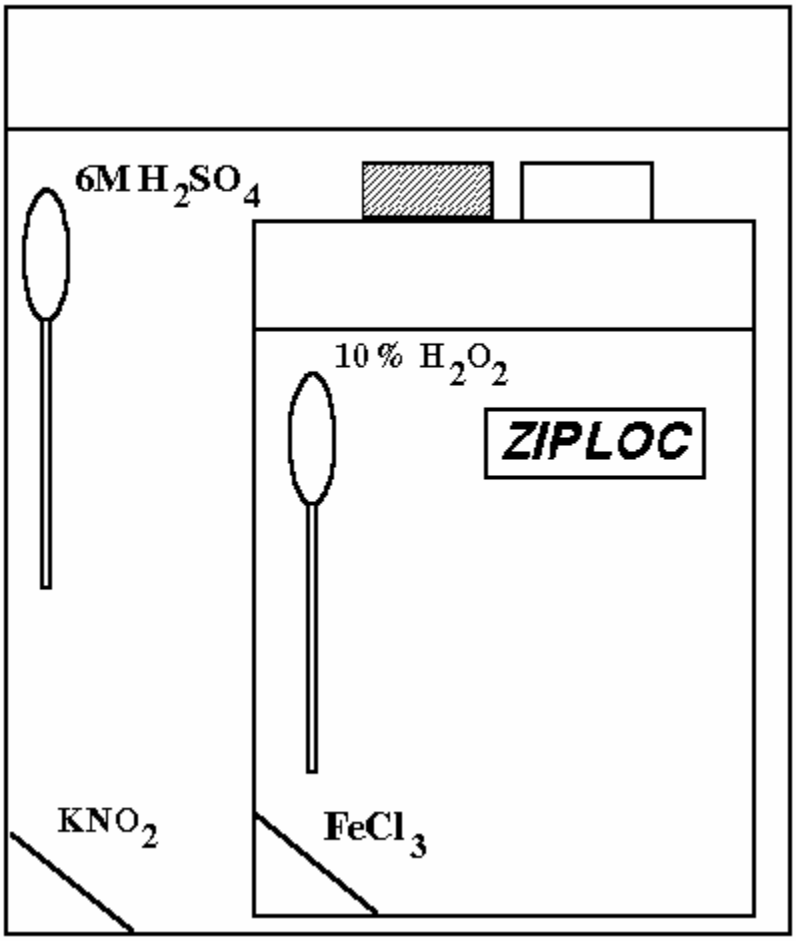
   The bag that will serve as a container for oxygen must be opened inside another bag. Utilize the bag with the OneZip™ closure for this procedure. See diagram on next page.

   a. Place about 1 gram of iron (III) chloride ($FeCl_3$) in the bottom corner of a 1-quart, heavy-duty Ziploc™ bag.
   b. Fill the appropriately labeled thin-stem, plastic pipet with 10% hydrogen peroxide (H_2O_2).

10% H_2O_2 is corrosive and will cause burns! Handle with care!

   c. Place the thin-stem, plastic pipet with 10% H_2O_2 in the plastic bag with the $FeCl_3$. Smooth out the bag so it contains a minimum amount of air, and then seal the bag. Take care not to press against the pipet.

3. Combination of the two generating bags
   a. Place the 1-quart bag (oxygen generating bag) inside the bag containing the thin-stem, plastic pipet with 6 M H_2SO_4 and KNO_2 (nitric oxide generating bag). Smooth out the bag so it contains a minimum amount of air, and then seal the bag. Take care not to press against the pipet. Your assembled system should look like the diagram on the following page.



b. Without opening either bag, generate the oxygen in the inner bag by dropping the H_2O_2 on the $FeCl_3$.
c. Generate NO by slowly squeezing the thin-stem, plastic pipet so that the 6 M H_2SO_4 slowly drops onto the KNO_2. When the reaction is complete, you should have a Ziploc™ bag that is partially filled with NO (and possibly some NO_2). Record your observations in the data table.
d. Now without opening the larger bag with NO, open the small bag with oxygen and observe the result and record your observations.
e. The larger bag now contains a mixture of nitrogen oxides which you can use for the tests below.

Technique for gas sampling from Ziploc™ bag:

1. To fill a pipet with NO gas, take a dry, thin-stem plastic pipet and squeeze the bulb to expel the air in it.
2. Slowly push the tip of the pipet against the seal of the plastic bag at one corner of the plastic bag containing the gas to be tested. With a bit of

practice, you will be able to push the pipet tip so that the seal just opens around it.
3. Push the pipet tip all the way into the bag, release the bulb so that the gas enters the pipet, and withdraw a sample of gas.
4. As the tip leaves the bag, reseal the bag along the "zip strip". This maintains the integrity of the gas in the bag.

III. Properties of sulfur dioxide and nitrogen dioxide

Use the method described in the Techniques section from Atmospheric Gases: Part One to perform the test with the SO_2 gas generated in this experiment. Use the gas sampling method described in the Techniques section above to obtain a pipet of NO gas to perform the test in this experiment.

A. Bubble the SO_2 gas into well C3 containing the tap water and universal indicator by slowly turning the handle to the parallel position (see Techniques Section in Atmospheric Gases: Part One).
B. By comparing colors with the buffer solutions, determine the pH of the dissolved gas in well C3.
B. Record your results in the data table.
C. Place the tip of the gas-filed (NO) pipet into well C4 containing tap water and universal indicator,
D. Bubble the gas through the solution. By comparing colors with the buffer solutions, determine the pH of the dissolved gas in well C4.
E. Record your results in the data table.

IV. Clean up

Bags containing the nitrogen oxides and flasks containing the sulfur dioxide must be cleaned in the fume hoods. Open the containers and allow all of the gases to be vented into the hood. Then carefully pour off any liquids remaining into the hood sink. Rinse all of the bags and flasks with tap water and dry the bags with paper towels. **Do not turn bags inside out to dry. Leave the flasks upside down on a paper towel on your lab bench away from the edge where they could get knocked over. Make sure to turn the valve to the closed position. Balloons will be changed as needed.** Rinse out the pipets with water and leave all the supplies for the next class. Be sure to wash hands well with soap and water before leaving.