Supplemental Experiment 1

Refrigerant Gases

> *Warning: do not do this experiment if you have a sensitivity to sulfites or have an asthmatic condition! Sulfites have been shown to cause severe asthma attacks in a few sensitive individuals.*

Introduction

Today it is hard to understand why the discovery of chlorofluorocarbons (CFCs) was hailed as one of the wonders of modern chemistry. They are now viewed with great concern because of their destructive effect on the stratospheric ozone layer. (See Chapter 2 in *Chemistry in Context* for more detail.) In order to better understand the attractiveness of CFCs, it is useful to examine the properties of two refrigerant gases, sulfur dioxide and ammonia, that were used before CFCs were developed. In this experiment you will prepare samples of both sulfur dioxide and ammonia, investigate some of their properties, and compare them with a current CFC-substitute compound.

Background Information

Refrigerators and air conditioners take advantage of the physical properties of liquids and gases. Heat energy is given off when a vapor condenses to a liquid. Conversely, heat energy is absorbed when a liquid vaporizes. The cooling mechanism in a refrigerator or air conditioner is a closed system containing a compound that boils at a low temperature, but can easily be converted back to a liquid under pressure. The liquid compound is allowed to vaporize in metal tubes inside the refrigerator. As the liquid vaporizes it takes heat from its surroundings, cooling the inside of the refrigerator. The vapor produced is pumped outside of the cold compartment where it is converted back to a liquid under pressure applied by a compressor. The vapor condenses in metal tubes outside of the refrigerator compartment. As the gas is converted back to a liquid it loses the heat it picked up inside the refrigerator. This explains why the metal coils on the back of most refrigerators always feel warm. In essence heat is pumped out of the refrigerator and into the room containing the refrigerator. In an air conditioner, the heat removed from the room air is pumped to the outside air.

To efficiently carry out this refrigeration cycle, refrigerant gases must have certain physical properties. They must have boiling points well below 0˚C so that they do not condense back to a liquid inside the refrigerator or freezer compartment, and they must be easily liquefied under pressure. Furthermore, they should be non-flammable. Before CFCs, the most common refrigerant gases that met these requirements were ammonia (NH_3) and sulfur dioxide (SO_2). Ammonia boils at –33°C and sulfur dioxide boils at –10˚C . Both can be liquefied at room temperature under moderate pressure. At 86˚C, ammonia requires a pressure about 11 times greater than atmospheric pressure and sulfur dioxide requires a pressure about 5 times greater than atmospheric pressure.

In this experiment you will utilize chemical reactions to make both ammonia gas and sulfur dioxide gas; then you will compare their properties to those of a modern refrigerant gas. Since CFCs are no longer available, you will use one of the HCFC or HFC gases that have replaced CFCs. (These are discussed in the text, section 2.19.)

Sulfur dioxide will be produced by means of the following reaction:

$$Na_2SO_3 + 2HCl \rightarrow 2NaCl + H_2O + SO_2$$

Ammonia will be produced by warming concentrated ammonium hydroxide solution:

$$NH_4OH_{(aq)} + \text{heat} \rightarrow H_2O + NH_3$$

You will compare the three refrigerant gases with respect to odor, solubility in water, reactivity with water, and reactivity with potassium permanganate solution. If a compound reacts with potassium permanganate it indicates that the compound is fairly reactive and can lose electrons easily, a process known as oxidation. Potassium permanganate ($KMnO_4$) is a deeply colored compound, but the reaction product ($MnSO_4$) is colorless; therefore it easy to observe whether a chemical reaction has occurred.

Overview of the Experiment

1. Construct a reaction vessel for generating sulfur dioxide.
2. Prepare a sample of sulfur dioxide and describe its odor.
3. Observe the reactions of sulfur dioxide with water and with potassium permanganate.
4. Generate ammonia gas and describe its odor.
5. Observe the reaction of ammonia gas with water and with potassium permanganate.
6. Collect a sample of refrigerant gas and describe its odor.
7. Observe the reaction of refrigerant gas with water and potassium permanganate.

Materials Needed

Chemicals	Equipment
• 1-2 grams of Na_2SO_3 • 2 mL of concentrated HCl • 2 mL of concentrated ammonium hydroxide • 1 mL of bromothymol blue • phenolphthalein solution • 2 mL dilute potassium permanganate solution • HCFC or HFC refrigerant gas	• one jumbo-jumbo15 mL plastic pipet • 3 graduated plastic pipets (5 mL) • 2 super-jumbo plastic pipets (8 mL) • graduated micro tip pipet (1.5 mL) • one thin stem transfer pipet (4 mL) • one #0 two hole rubber stopper • plastic teaspoon • wellplate • three 50-mL beakers • one 250-mL beaker • thermometer • scissors

Note that five different sizes of plastic pipets are needed for this experiment. They are shown in the following diagram*(Figure 1)*.

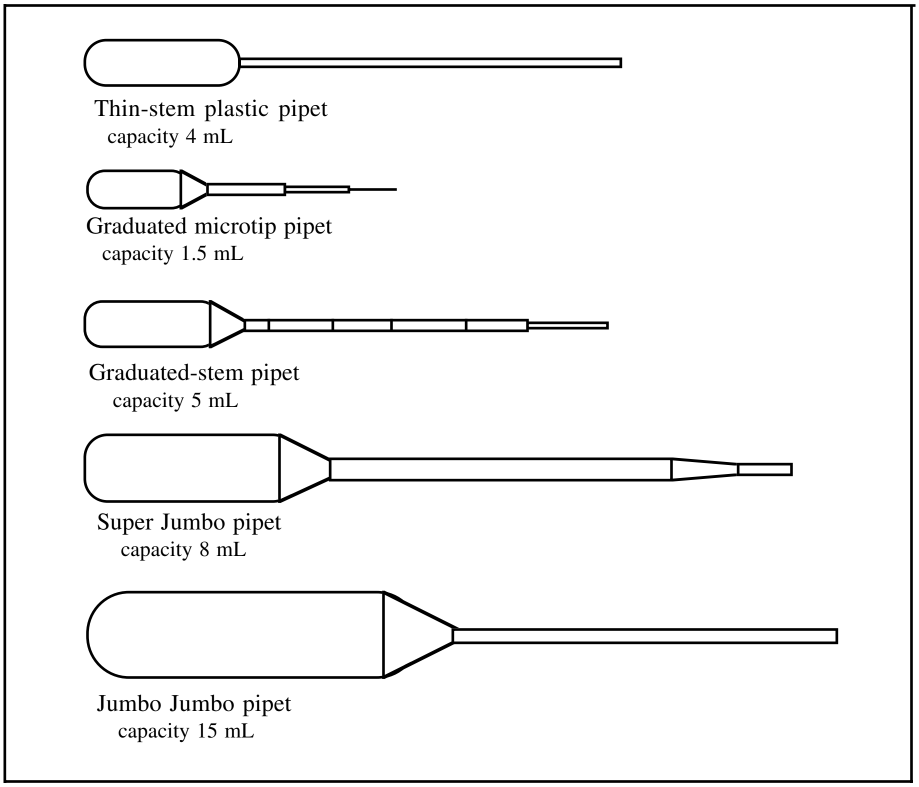

Figure 1 The types of transfer pipets used in this experiment.

SAFETY NOTES:

- Both of the liquids used in this experiment, concentrated hydrochloric acid (HCl) and concentrated ammonium hydroxide (NH_4OH), are corrosive materials and should be handled cautiously.
- Both of the gases produced in this experiment, ammonia (NH_3) and sulfur dioxide (SO_2), are very toxic and should not be allowed to escape into the lab.
- See warning on previous page about individual sensitivity to sulfites or an asthmatic condition.

EXPERIMENTAL PROCEDURE

I. Constructing a Reaction Vessel for Sulfur Dioxide

1. Cut off the bulb section of a super-jumbo pipet just after the stem widens to the size of the bulb (*Figure 2*).

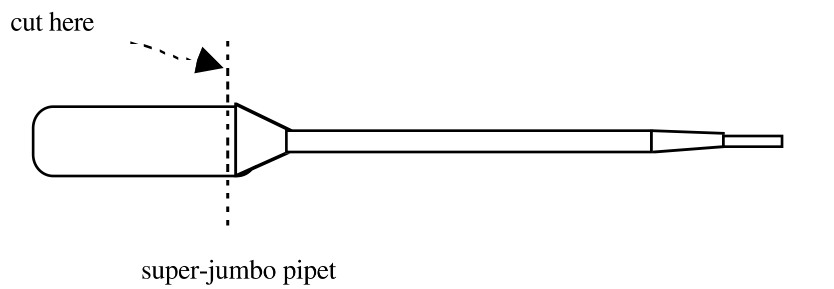

Figure 2 Where to cut a super jumbo pipet.

2. Stand the cut off bulb in a well of a wellplate.

3. Cut the stem off of a 15 mL jumbo-jumbo transfer pipet *(Figure 3)*. Discard the bulb end.

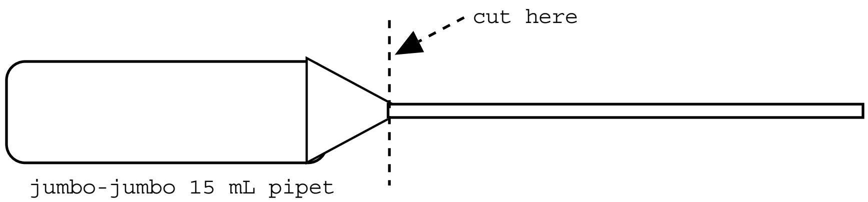

Figure 3 Where to cut a jumbo-jumbo pipet.

4. Obtain a # 0 two hole rubber stopper. Push the stem of the 15 mL transfer pipet through one hole and a graduated micro-tip pipet through the other hole *(Figure 4)*.

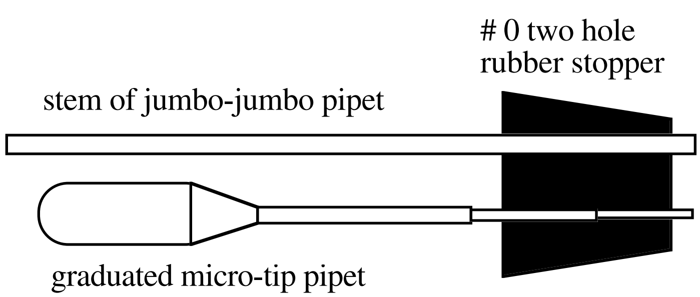

Figure 4 The top of the reaction vessel.

5. The stopper assembly should fit tightly into the bulb section of the transfer pipet prepared in step 1 *(see Figure 5)*. The reaction vessel will just fit into a well in a 24-well wellplate *(Figure 6)*.

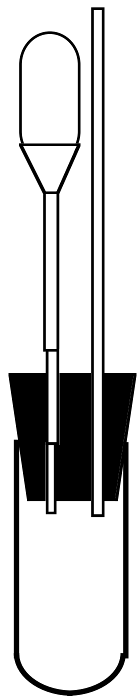

Figure 5 The reaction vessel

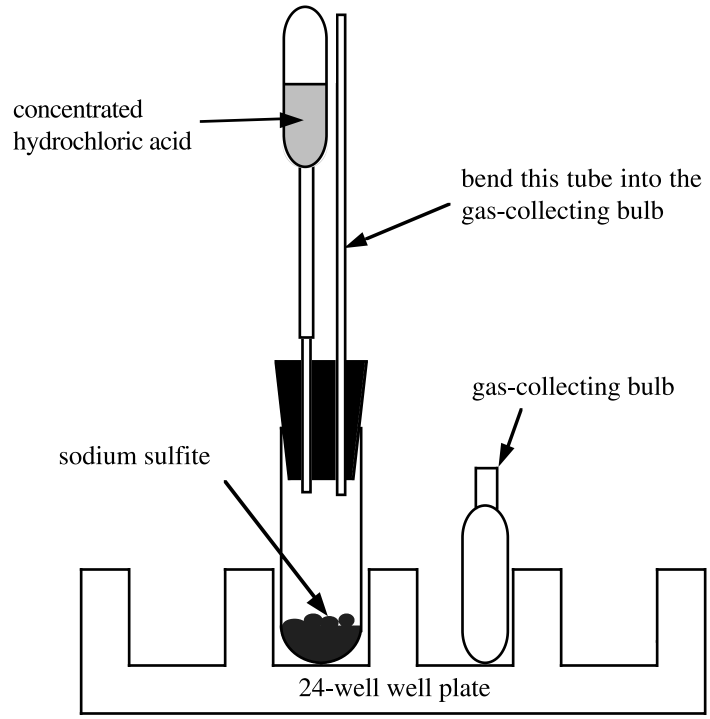

Figure 6 The complete reaction set-up for generating sulfur dioxide.

II. Preparing and Testing Sulfur Dioxide

1. Place 1 gram of sodium sulfite in the bottom of the reaction vessel and place the vessel in a well of a wellplate.
2. Remove the small pipet and half fill it with concentrated hydrochloric acid. Then put it back in the stopper.
3. Cut off the bulb of a graduated-stem pipet at the 1 mL mark. This will serve as the container to trap the sulfur dioxide. Place this bulb in a well next to the reaction vessel. The entire assembly should look like *Figure 6.*
4. To generate sulfur dioxide, bend the straight pipet stem so that the end goes into the gas-collecting pipet bulb. Take the assembly over to the fume hood. Now squeeze the bulb of the pipet containing hydrochloric acid so as to add 2-3 drops to the sodium sulfite. Sulfur dioxide gas will be produced and will carry over into the gas-collecting bulb.

A. Determining the Solubility of Sulfur Dioxide in Water

1. Add about 40 mL of water to a 50-mL beaker. In the fume hood generate and collect some sulfur dioxide by the method described in step 4 above.
2. To test the solubility of sulfur dioxide in water, remove the straight stem from the gas-collecting bulb filled with sulfur dioxide and quickly invert the bulb into the beaker of water so that the

opened end is below the surface of the water. Observe for a minute or two. If water comes up into the bulb, this means the sulfur dioxide is soluble in water. Record your observations.

3. Rinse out the gas collecting bulb with water.

B. Reactions of Sulfur Dioxide with Water and with Potassium Permanganate

1. Put a few drops of water and a drop of bromothymol blue indicator into the gas-collecting pipet bulb. Bend the straight pipet stem so that the end goes into the gas-collecting bulb.
2. Take your assembly over to a fume hood. Generate some sulfur dioxide by adding 2-3 drops of HCl (as described above). Shake the gas-collecting bulb gently and record any changes in the color of the indicator. Bromothymol blue is yellow in acid and blue in base.
3. Remove the gas-collecting bulb and rinse it out with water. Then add a few drops of pink potassium permanganate solution. Re-insert the tube from the reaction vessel and again generate some sulfur dioxide by adding 2 or 3 more drops of concentrated hydrochloric acid to the sodium sulfite. Gently shake the collecting bulb and record your observations.
4. If you have not already gotten a whiff of sulfur dioxide, *very gently* wave your hand past the opening of the pipet stem in the direction of your nose. Do not put your nose near the pipet stem and take a deep breath. Sulfur dioxide is a toxic gas and you should not inhale very much of it**.** Describe the odor of the gas on your data sheet**.**

C. Clean Up for Part II.

1. Put 50 mL of water in a 250-mL beaker. Remove the pipet with concentrated hydrochloric acid from the reaction vessel and slowly add the acid to the 50 mL of water.
2. Neutralize the acid by adding solid sodium bicarbonate a little at a time until it no longer produces bubbles when added. At this point the acid has been neutralized and the mixture can be safely poured down the drain. The potassium permanganate solution should be placed in the waste container provided.
3. Pour the sodium sulfite-acid mixture from the bottom of the reaction vessel into the designated waste container. All of the plastic parts can be rinsed with water and discarded unless your instructor asks you to save them. Save the rubber stopper for the next class.

III. Preparing and testing Ammonia

1. In the fume hood use a hot plate to heat 30 mL of water in a 100 mL beaker to 65˚C.
2. Half fill a thin stem transfer pipet with concentrated ammonium hydroxide. The stem of the pipet must not contain any liquid. If liquid is in the stem, force it to the top by gently squeezing the pipet bulb and blot off the liquid with a paper towel.
3. Put 50 mL of water and three drops of phenolphthalein solution in a small beaker.
4. Cut off the bulb of a graduated transfer pipet to use as a gas-collection bulb. Place it over the stem of the transfer pipet half filled with ammonium hydroxide. *(see Figure 7).*

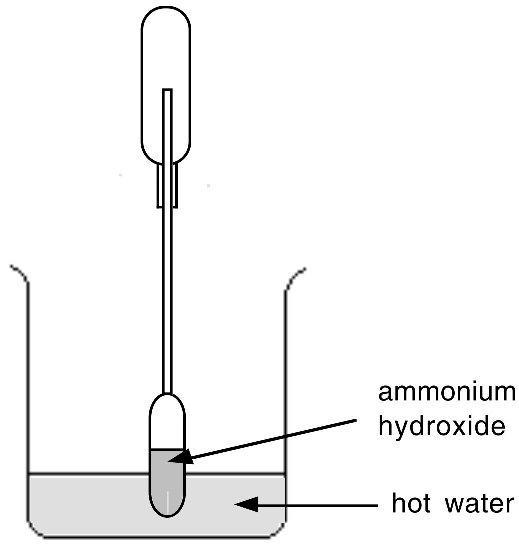

Figure 7 The set up for generating and collecting ammonia gas

5. In the hood, dip the end of the bottom pipet (half filled with ammonium hydroxide) into the hot water. After a few seconds the ammonium hydroxide should begin to bubble as ammonia gas is driven off from the warm ammonium hydroxide solution. After 25-30 bubbles appear, your gas-collection bulb should be full of ammonia gas.
6. Quickly remove the collection bulb, keeping the open end pointed down. Touch the opened end of the collection bulb to the phenolphthalein solution so that the open end of the bulb is about an eighth of an inch below the surface of the liquid. Gently move the bulb from side to side while the opened end is in the liquid. Describe what you observe. Phenolphthalein is colorless in acid and red in base
7. Place 40 mL of a pink potassium permanganate solution in a 50-mL beaker. Repeat steps 5-6 substituting the potassium permanganate solution for phenolphthalein. Record your observations on the data sheet.
8. If you have not already gotten a whiff of ammonia, *gently* wave your hand past the opening of the pipet stem in the direction of your nose. Do not put your nose near the pipet stem and take a deep breath. Ammonia is a toxic gas and you should not inhale very much of it**.** Describe the odor of the gas on your data sheet**.**

Clean Up for Part III.

1. Empty the excess ammonium hydroxide solution into the waste disposal container provided in the fume hood.
2. The phenolphthalein can be washed down the drain with a large amount of water. The potassium permanganate solution should be placed in the waste container provided

IV. Testing a Modern Refrigerant Gas

These tests will be done with one of the substances currently available as replacements for CFCs. It will most likely be an HCFC (such as HCF_2Cl) or an HFC (such as $H_2C_2F_4$). Your instructor will demonstrate how to obtain small amounts of gas from the pressurized container.

1. Cut off the stem of a graduated pipet to make another gas-collection bulb.
2. Fill the dry gas-collecting bulb with refrigerant gas, keeping the opened end of the bulb pointed up. Test the gas sample for solubility in water by inverting the collection bulb in a beaker of water just as you did with sulfur dioxide and ammonia.
3. Test reactivity of the gas with water by putting a few drops of bromothymol blue indicator solution in the gas-collecting bulb. Fill the bulb with refrigerant gas and shake the bulb. Record your observations. Rinse out the gas-collecting bulb with water.
4. Test the reactivity of the gas with potassium permanganate solution by putting a few drops of potassium permanganate solution in the gas-collecting bulb. Fill the bulb with refrigerant gas and shake the bulb. Record your observations.

Clean Up for Part IV.

The phenolphthalein can be washed down the drain with a large amount of water. The potassium permanganate solution should be placed in the waste container provided.

Questions To Be Answered After Completing This Experiment

Write out answers to the following questions on a separate sheet of paper and hand it in along with the data sheet.

1. Judging from your test results, which gas is the most soluble in water? What observations lead you to this conclusion?
2. What do the test results from reactions with water and potassium permanganate tell you about the chemical properties of the gases tested?
3. If ammonia and sulfur dioxide were used in household refrigerators, what warning labels would you want to see attached to the refrigerators? Write out the proposed labels.
4. On the basis of your observations, why do you think that CFCs were considered a boon to civilization when they were discovered?
5. Some large commercial refrigeration units still use ammonia as a refrigerant gas. Why do you think ammonia is still being used?
6. Challenge: When you were collecting the gases, why do you think you were instructed to hold the collection bulb with its open end down to collect ammonia, but the open end up to collect sulfur dioxide and the refrigerant gas?

Name ____ Date ____________

Partner ____ Lab Section ________

Data Sheet – Refrigerant Gases

1. Properties of Sulfur Dioxide

Test	Observations
Solubility in water	
Reaction with water and indicator	
Reaction with Potassium permanganate solution	
Odor	

2. Properties of Ammonia

Test	Observations
Solubility in water	
Reaction with water and indicator	
Reaction with potassium permanganate solution	
Odor	

(continued next page)

Name ____ Date _____________

Partner ____ Lab Section _______

Data Sheet – Refrigerant Gases, page 2

3. Properties of a Refrigerant Gas

Name and chemical formula of the gas used: ____________________________________

Test	Observations
Solubility in water	
Reaction with water and indicator	
Reaction with potassium permanganate solution	
Odor	