

# Preparation and Properties of Nonmetals and Their Compounds

## Part One: Preparation of Oxygen, Nitrogen, Bromine and Iodine

There are seven naturally occurring diatomic molecules. In part one we will prepare four of them. We will then carry out some tests that will help us learn about their properties. During the experiment you will need a water bath (600mL beaker on a hot plate) to warm your solutions. Set it up now because water takes time to heat up.

### 1. Oxygen, O<sub>2</sub>

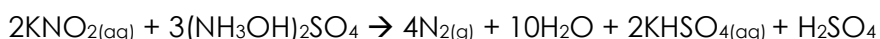
We will prepare oxygen from the decomposition of hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>). Hydrogen peroxide is easily decomposed by using manganese dioxide as a catalyst. Add about one mL of 3% hydrogen peroxide to a test tube. Pick up a very small amount of manganese dioxide on the tip of a spatula and drop it into the test tube. Record your observations. Moisten a piece of red litmus paper and hold it over the test tube - does it change color? Do the same test with blue litmus paper. Does it change color? Record your observations. Wave your hand over the top of the test tube to determine if there is an odor. Light a wooden splint and shake it until it goes out but still glows. Insert the glowing splint into the test tube but do NOT allow it to contact the solution - suspend it in the gas. Does oxygen support combustion? Is oxygen acidic or basic or neutral? Does oxygen have an odor?

**Note: When you smell any chemical compound wave your hand towards your nose carefully. Do not place test tubes under your nose and inhale deeply. This is dangerous.**

If the test tube contents stop producing bubbles before you've finished your test just pour out the contents of the test tube, rinse it, and repeat the procedure to produce more oxygen.

### 2. Nitrogen, N<sub>2</sub>

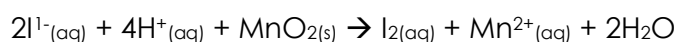
Nitrogen is very chemically stable and doesn't react easily so it is mostly identified by what it doesn't do. We will prepare nitrogen by the following reaction:



Place 1mL of potassium nitrite and 1mL of hydroxylamine sulfate in a test tube. You should observe gas bubbles. Carry out the same tests (litmus test, odor test, combustion test) you did for oxygen and note any differences in the results.

### 3. Iodine, I<sub>2</sub>

Of all the halogens iodine is the safest to prepare. We will begin with a source of iodide ion. This can be either potassium iodide or sodium iodide solution. Determine which of these is available to you. We will use 6M hydrochloric acid and a little manganese dioxide. This time manganese dioxide is a reactant not a catalyst. This is the reaction we will carry out:



Can you identify what atom is being reduced and what atom is being oxidized? At room temperature iodine is a solid that vaporizes easily and does not dissolve in water very well. This is because it is a non-polar molecule. It will dissolve in a non-polar solvent such as hexane (C<sub>6</sub>H<sub>14</sub>). We

will use a technique called extraction to bring the iodine from the water to the hexane. (**Note: Hexane is flammable!**)

Put two drops of 1M KI into a test tube. Add six drops of 6M HCl and a small amount of MnO<sub>2</sub> (use the tip of the spatula). Gently mix the contents of the test tube and observe any color changes. Put the test tube into a hot water bath. After a few minutes you should be able to observe some purple vapor coming from the test tube. If you do not observe purple vapor add two more drops of potassium iodide. Carefully test for any odor. Carry out the litmus paper test also but do **NOT** test for the support of combustion because of the flammability of hexane. As soon as the purple vapor reaches the top of the test tube remove the test tube and add 10 mL of distilled water. Stopper the test tube and shake it. Do you see any color changes? Pour about half of the liquid into a fresh test tube **leaving behind any unreacted solids**. Add 3mL of hexane to the liquid. Once again stopper and gently shake the test tube. Record the color of the hexane layer.

#### 4. Bromine, Br<sub>2</sub>

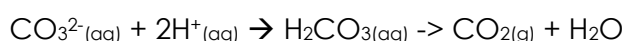
Now that you've had some practice working with halogens we will repeat this entire procedure but we will substitute bromide ion for the iodide ion and so we will produce bromine gas. Use sodium bromide and carry out the same steps as in the preparation of iodine. When you perform a test for an odor be very careful - do not inhale deeply. Bromine gas is irritating to the mucus membranes of the nose. You should be able to detect the odor with a minimal exposure to this gas. Carry out the litmus paper test also but do **NOT** test for the support of combustion because of the flammability of hexane.

In your laboratory notebook create a chart to summarize all of your observations about these nonmetals.

### Part Two: Preparation of CO<sub>2</sub>, SO<sub>2</sub>, NO and NO<sub>2</sub>

#### 1. Carbon Dioxide, CO<sub>2</sub>

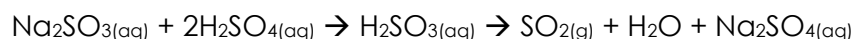
To produce carbon dioxide we will add an acid to the carbonate ion. The reaction produces carbonic acid and this will decompose into carbon dioxide and water. Here is the reaction:



Put one mL of 1M sodium carbonate in a small test tube and add six drops of 3M sulfuric acid. Test the gas for any odor. Place a glowing splint into the test tube to determine if carbon dioxide supports combustion. Test the gas for any acid/base properties using the litmus paper. Record all of your observations carefully.

#### 2. Sulfur Dioxide, SO<sub>2</sub>

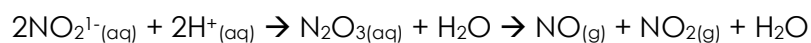
Sulfur dioxide is prepared in much the same way as carbon dioxide but it is much more soluble in water so you may not see any bubbles at room temperature. To decrease the solubility of sulfur dioxide place the test tube in a hot water bath. While it is in the bath use the litmus paper to test any vapor for evidence of an acid. Sulfur dioxide is a very acidic gas.



**Observe any odors but do not inhale this gas directly.**

### 3. Nitrogen dioxide, NO<sub>2</sub>, and Nitric Oxide, NO

The nitrite ion reacts with acid to form N<sub>2</sub>O<sub>3</sub> which then decomposes into nitric oxide and nitrogen dioxide. Here is the reaction:



To 1 mL of 1 M potassium nitrite in a small test tube add six drops of 3M sulfuric acid. Swirl the mixture. Observe any color changes. Warm the test tube. Continue to observe. Test the gases for the ability to support combustion and for any acid/base properties. Summarize the properties of these nonmetallic oxides in your laboratory notebook.

### Part Three: Preparation of NH<sub>3</sub> and H<sub>2</sub>S

#### 1. Ammonia, NH<sub>3</sub>

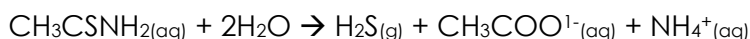
It is easy to confuse ammonia and ammonium hydroxide. They are related compounds. Ammonia is a gas and ammonium hydroxide is the compound that forms when this gas dissolves in water. As the concentration of ammonium hydroxide increases in a solution more and more ammonia will be liberated. We will make ammonium hydroxide and warm it - causing it to decompose into ammonia and water.



Add 1 mL of 1 M ammonium chloride (our source of ammonium ions) to a small test tube. Add 1 mL of 6M sodium hydroxide. Swirl the mixture and carefully test for any odor. Warm the test tube in the water bath and test for odor. When the odor is detected test the evolving gas for its ability to support combustion and for its acid/base properties.

#### **THIS LAST PREPARATION MUST BE CARRIED OUT IN THE HOOD!!**

Hydrogen sulfide is toxic. It also has a strong odor so we can detect it in very low concentrations. We will prepare a small amount of this gas **UNDER THE HOOD**. We will decompose thioacetamide as follows:



Use one mL of 1M thioacetamide in a small test tube and add six drops of 3M sulfuric acid. Put the test tube in a boiling water bath for one minute. Test the vapors for acid/base properties and for the ability to support combustion.

### Part Four: Identity of an Unknown Solution

Your unknown will not be a gas - it will be one of the solutions you used to make these gases. It will not be an acid or a base. You need to try to use your unknown to prepare a known gas. Possible unknowns are 3% H<sub>2</sub>O<sub>2</sub>, NH<sub>4</sub>Cl, NaBr, KI, Na<sub>2</sub>CO<sub>3</sub>, Na<sub>2</sub>SO<sub>3</sub>, KNO<sub>2</sub>. Report the identity of your unknown.

### References

This lab is a modified version of Experiment 18: Some Nonmetals and Their Compounds - Preparation and Properties from *Chemical Principles in the Laboratory* 10th Ed. by Slowinski, Wolsey and Rossi.

## Set Up for Preparation and Properties of Nonmetals and Their Compounds

### Chemical Compounds (prepare 4 of each of the following)

#### 50mL dropping bottles

freshly prepared 3% hydrogen peroxide (unstable after one week)

1M potassium iodide

1M sodium bromide

#### salt solutions - 200mL bottles

1M ammonium chloride

1M potassium nitrite

1M hydroxylamine sulfate

1M sodium carbonate

1M thioacetamide

1M sodium sulfite (must be fresh)

#### acids and bases - 200mL bottles

3M sulfuric acid

6M hydrochloric acid

6M acetic acid

6M sodium hydroxide

6M ammonium hydroxide (please label as ammonium hydroxide and not ammonia)

#### organic - 200mL bottle

hexane

#### solids

50 g bottles manganese dioxide

### Supplies for entire class of 32 students working in pairs

4 vials red litmus paper

4 vials blue litmus paper

4 containers of wooden splints

8 hot plates for water baths

8 400mL beakers for water baths

4 spatulas for bottles of manganese dioxide

150 test tubes with matching size corks and small pieces of paraffin film to cover corks

### Unknowns

*Each student receives 10mL of one of the following:*

3%  $\text{H}_2\text{O}_2$  (must be fresh, store in refrigerator), 1M  $\text{NH}_4\text{Cl}$ , 1M  $\text{NaBr}$ , 1M  $\text{KI}$ , 1M  $\text{Na}_2\text{CO}_3$ , 1M  $\text{Na}_2\text{SO}_3$  (must be fresh), 1M  $\text{KNO}_2$