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. The water baths must be set up under the hoods. There are four hoods. Each hood holds two water baths - one on each side.

When working at the hood the MAXIMUM number of students at the window should never exceed four (4). Some of these preparations are done under the hood - others may be done in the open. You do NOT have to do them in order. If the hood is too busy - skip to another preparation.

1. Oxygen, O_2 - Done in the open.
We will prepare oxygen from the decomposition of hydrogen peroxide (H_2O_2). Hydrogen peroxide is easily decomposed by using manganese dioxide as a catalyst. Add **about** one mL of 3% hydrogen peroxide to a test tube. (*The word about is used when the exact amount is not important.*) 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 with distilled water 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?

These are typical tests done to identify a gas. You will repeat these tests on other gases unless instructed NOT to do a certain test.

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_2 - Done in the open
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:

$$2KNO_{2(aq)} + 3(NH_3OH)_2SO_4 \rightarrow 4N_{2(g)} + 10H_2O + 2KHSO_{4(aq)} + H_2SO_4$$

Place about 1mL of potassium nitrite and about 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_2 - Done Under the Hood!!

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:

$$2I^{1-}_{(aq)} + 4H^{+}_{(aq)} + MnO_{2(s)} \rightarrow I_{2(aq)} + Mn^{2+}_{(aq)} + 2H_2O$$

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_6H_{14}) or heptane (C_7H_{16}). We will use a technique called extraction to bring the iodine from the water to the hexane. (**Note: Hexane is flammable! It needs to be kept away from any flame or hot plate. It doesn't need a spark to catch on fire - if it gets hot enough it will ignite. When you use it be sure to put the lid back on the bottle so the vapor doesn't escape any more than what is unavoidable. The hexane should be placed on the lab bench and NOT in the hood where the hot plates are located.**)

UNDER THE HOOD:

Make sure you bring the 10mL of water that you will need to add to the test tube with you.

Put two drops of 1M KI into a test tube. Add six drops of 6M HCl and a small amount of MnO_2 (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. You may remove the test tube from the water bath to perform tests but LEAVE IT UNDER THE HOOD! Carry out the litmus paper test also but do **NOT** test for the support of combustion. 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 invert it gently. Do you see any color changes? At this point the iodine vapor is in the water. You may now remove the test tube from the hood and continue the procedure.

OUTSIDE OF THE HOOD:

Pour about half of the liquid into a fresh test tube **leaving behind any unreacted solids**. Add 3mL of hexane to the liquid. Stopper and shake the test tube. You will need to give it a good shake. If you hold the test tube down in the sink while you shake this will ensure that you do not lose the stopper. You need to keep your thumb over the stopper while you shake. If you are not sure how to do this - ask the instructor to show you. **Record the color of the hexane layer. The iodine is in this layer now.**

Dispose of the hexane mixture in the ORGANIC WASTE receptical.

4. Bromine, Br_2

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.

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

Part Two: Preparation of CO_2, SO_2, NO and NO_2

1. Carbon Dioxide, CO_2 - Done in the open.

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

$$CO_3^{2-}{}_{(aq)} + 2H^+{}_{(aq)} \rightarrow H_2CO_{3(aq)} \text{ -> } CO_{2(g)} + H_2O$$

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_2 - Done in the hood.

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.

$$Na_2SO_{3(aq)} + 2H_2SO_{4(aq)} \rightarrow H_2SO_{3(aq)} \rightarrow SO_{2(g)} + H_2O + Na_2SO_{4(aq)}$$

Observe any odors but do not inhale this gas directly.

3. Nitrogen dioxide, NO_2, and Nitric Oxide, NO - Done in the hood.

The nitrite ion reacts with acid to form N_2O_3 which then decomposes into nitric oxide and nitrogen dioxide. Here is the reaction:

$$2NO_2^{1-}{}_{(aq)} + 2H^+{}_{(aq)} \rightarrow N_2O_{3(aq)} + H_2O \rightarrow NO_{(g)} + NO_{2(g)} + H_2O$$

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_3 and H_2S

1. Ammonia, NH_3 - Done in the open.

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.

$$NH_4^{1+}{}_{(aq)} + OH^{1-}{}_{(aq)} \rightarrow NH_4OH_{(aq)} \rightarrow NH_{3(g)} + H_2O$$

Add 1 mL of **1 M ammonium chloride** (our source of ammonium ions) to a small test tube. Ammonium chloride solution does not have any odor. 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. You will definitely smell ammonia.

THIS LAST PREPARATION WILL BE DONE AS A DEMONSTRATION OR MAY BE OMITED.

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:

$$CH_3CSNH_{2(aq)} + 2H_2O \rightarrow H_2S_{(g)} + CH_3COO^{1-}{}_{(aq)} + NH_4^{+}{}_{(aq)}$$

One mL of 1M thioacetamide is placed into a small test tube and six drops of 3M sulfuric acid are added. The test tube is placed in a boiling water bath for one minute. The vapors may be tested for acid/base properties and for the ability to support combustion. The smell of hydrogen sulfide is VERY strong.

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_2O_2, NH_4Cl, NaBr, KI, Na_2CO_3, Na_2SO_3, KNO_2**. You will attempt various preparations with your unknown to see what works. Remember not to add the chemicals in this list - you are trying to find out if your unknown is one of these substances.

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 (1 bottle only for demonstration)
1M sodium sulfite (must be fresh)

acids and bases - 200mL bottles
3M sulfuric acid
6M hydrochloric acid
6M sodium hydroxide

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 set up under the hoods
8 - 400mL beakers for water baths
4 spatulas for bottles of manganese dioxide
150 test tubes
40 rubber stoppers (size 00 and 0)

Unknowns

Each student receives 10mL of one of the following:
3% H_2O_2(must be fresh, store in refrigerator), 1M NH_4Cl, 1M NaBr, 1M KI, 1M Na_2CO_3, 1M Na_2SO_3 (must be fresh), 1M KNO_2