CHM 115 Lab 10

Gaseous Equilibrium: Some Properties of NO_2 Gas

In this lab work you will synthesize nitrogen dioxide, study its equilibrium with dinitrogen tetroxide, and learn something about its properties.

Many of the materials that you will be working with in this lab are hazardous. You must exercise care not to injure yourself or others. The NO_2 and the N_2O_4 gases are toxic so all work with them must be done under the fume extractor or in a fume hood. Concentrated nitric acid, HNO_3, can cause serious burns if it gets on your skin or clothing. Plastic gloves are to be worn when working with these chemicals. In this lab you will cut and bend glass tubing. Working with glass tubing can result in a cut or burn if the correct procedures are not followed. As always, wear your eye protection. Make certain you know where the safety equipment is located in the lab and how it is used in case of an emergency.

In part A, NO_2 gas is produced by reacting concentrated nitric acid and copper metal in a generator that you will construct. Some of the gas will be collected and studied.

$$Cu(s) + 4HNO_3\ (aq) \rightarrow Cu(NO_3)_2\ (aq) + 2NO_2(g) + 2H_2O$$

Part B pertains to a study of gaseous equilibrium. Nitrogen dioxide gas exists in equilibrium with dinitrogen tetroxide, its dimer, as shown by the equation:

$$2\ NO_2(g) \leftrightarrows N_2O_4(g) \qquad \Delta H = \text{negative value}$$

You are to study the influence of temperature on the position of equilibrium for this reaction. Since NO_2 gas is brown and N_2O_4 is colorless, the concentration of NO_2 in a closed tube can be determined by measuring the amount of light absorbed by the gas (i.e. determine Abs using a Spectronic 20). A tube of the gas mixture, after heating for several minutes in boiling water, will be placed in the Spectronic 20, then allowed to cool to room temperature and the Abs measured at specified time intervals. The tube will then be cooled in the ice bath, placed in the Spec 20 and Abs measured as the mixture warms to room temperature. The amount of light transmitted through the sample is related to absorbance (a unitless value) by A = 2 - log(%T). And absorbance (A) is directly proportional to concentration of the colored molecules (this is Beer's Law). From plots of the two series of Abs values vs time you will be able to gain a better understanding of the influence of temperature on the position of equilibrium.

PART A: The Synthesis of Nitrogen Dioxide

WEAR SAFETY GOGGLES, and work in pairs.

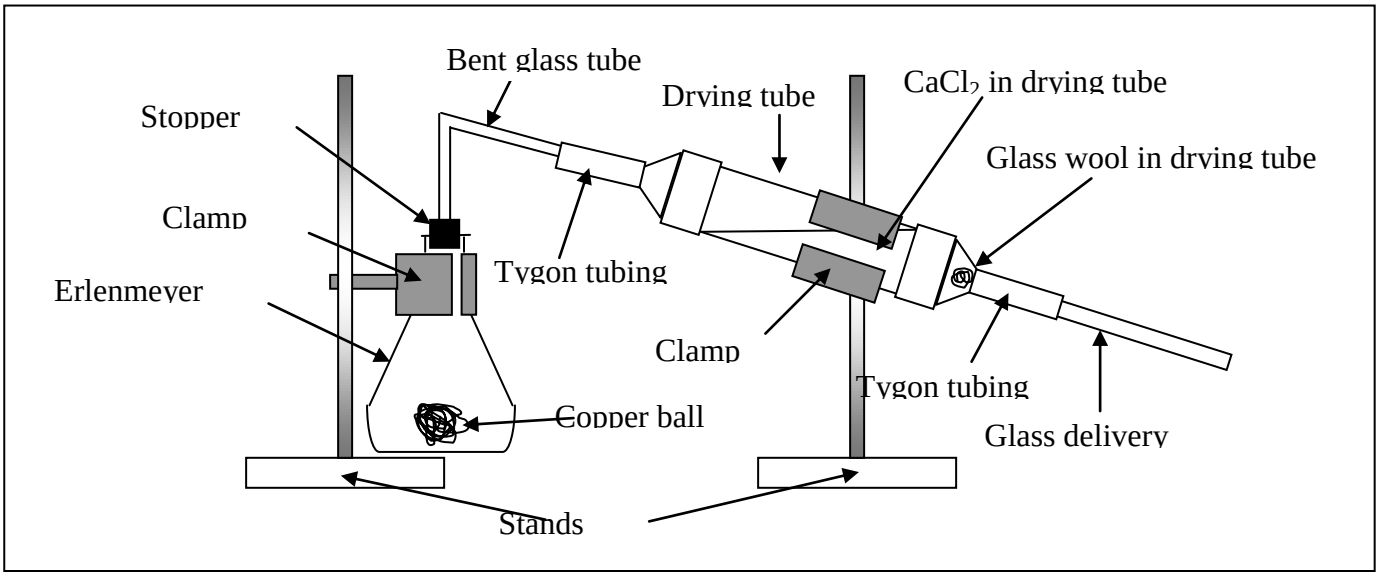
1. Take a 5" long piece of glass tubing and bend it at about a 60° angle. Allow to cool. Fire polish both ends of the bent piece. Allow to cool.

2. Hold the bent tube in a towel and push one end of the bent tubing through the one-holed stopper provided. Great care is required! Your instructor will demonstrate this operation before you do it.

3. Place a small glass wool plug into the internal narrow tube of a DRY drying tube.

4. Tip some granular calcium chloride onto a piece of paper towel and pour the solid into the drying tube until it is about 1/2 full.

5. Set up the following reaction apparatus for the synthesis. Position fume extractors at both ends of the set-up equipment. Make certain that the apparatus is clamped firmly in place and that the fume extractors are securely positioned.



6. One of you, put on plastic gloves. Steps that have an ASTERISK should be carried out by the person with gloves on.

*7. Remove the stopper to the 125 mL Erlenmeyer flask. Pour 15 mL of concentrated nitric acid (HNO_3) into the flask (use a graduated cylinder).

*8. Place several small pieces of copper metal into the flask. Replace the stopper firmly. The copper will begin to dissolve and produce brown gas. Put the delivery tube on the reaction apparatus almost all the way into one screw cap tube. Allow the NO_2 to sweep out the air from the apparatus and tube. *Be careful not the inhale the brown gas!*

*9. Remove the tube and quickly screw on the cap; place in the test tube rack. (Take off your plastic gloves!)

10. Set up a Bunsen burner apparatus and fill a 250 mL beaker (to within about 1/2" of top) with water. Boil the water.

11. Make an ice bath by filling a beaker with ice (2/3) and water (1/3), and go straight to part B.

PART B: Investigation of the dimerization reaction of Nitrogen Dioxide

1. Set the wavelength control of the spectrophotometer to 500 nm. Set zero. Place the empty tube into the sample compartment. Set 100% T.

2. Remove the empty tube and place the tube containing the brown NO_2 into the sample compartment. Slowly change the wavelength control from 520 to 480 nm. Note the wavelength at which the %T reads about 25%. Record this wavelength in your lab notebook. Set the instrument at the wavelength you chose and re-zero (use the empty tube!). Change the Spec 20D to Absorbance mode after calibrating.

3. Place the spectrophotometer tube containing the NO_2 into the boiling water bath. Hold it in as far as possible with two (2) test tube clamps. Leave the tube in the boiling water for about 4-5 minutes. Take the temperature of the boiling water.

4. Remove the tube, quickly dry it with a paper towel, and place the tube into the sample compartment of the spectrophotometer, which you set and zeroed (in %T mode).

5. Quickly take absorbance readings versus time, i.e. take Abs reading every 5 seconds for the first minute, then every 10 or 20 seconds for another 3 or 4 minutes (not longer).

6. Remove the tube from the sample compartment and place it in the ice bath (as far as possible). Leave it in the ice bath for about 10 minutes. At about 6-9 minutes, re-zero the spectrophotometer (empty tube) in %T mode and check the wavelength setting. Take bath temperature.

7. Remove the tube and quickly dry it with paper towel, and place it in sample compartment.

8. Take absorbance readings versus time, as in step 5. Remember to change the spectrometer to Absorbance mode before taking measurements.

9. In the fume hood, remove the plastic stopper from the spectrophotometer tube, pour out the NO_2/N_2O_4, and rinse the tube with water.

10. Plot a graph of absorbance versus time (time on x- axis) for both hot and cold tubes, on the same graph.