Experiment 1

MOLAR VOLUME OF NITROGEN GAS

Objective

The purpose of this experiment is to determine the molar volume of nitrogen gas under standard temperature and pressure (STP) by utilizing the reaction between sulfamic acid and sodium nitrite and the ideal gas law.

Lab techniques

- Weighing chemicals
- Measuring volume, pressure, and temperature

Introduction

The mole, symbol mol, is the SI unit of amount of substance. One mole contains exactly $6.02214076 \times 10^{23}$ elementary entities. This number is called the Avogadro number. One mole of hydrogen gas (H_2) and one mole of nitrogen gas (N_2) both contain 6.022×10^{23} molecules but have different molar masses, *i.e.* 2.016 g and 28.02 g, respectively.

The term molar volume refers to the volume occupied by one mole of a given substance. For an ideal gas at STP (*i.e.* 0°C and conventionally 1 atm; International Union of Pure and Applied Chemistry, IUPAC, has re-defined the standard pressure as 1 bar, but 1 atm is still often used), the molar volume is 22.414 L.

In general, a real gas at STP also behaves closely to an ideal gas. It is because the gaseous particles are far apart and hence exert negligible force on each other, and the volume occupied by the gaseous particles themselves can be ignored (considered as 0) compared with the volume of the container. Therefore, the ideal gas equation 1-1 and 1-2 can also be applied to hydrogen and nitrogen gases, giving their molar volumes at STP very close to 22.414 L.

$$PV = nRT \qquad (1\text{-}1)$$

$$\frac{P_1 \times V_1}{n_1 \times T_1} = \frac{P_2 \times V_2}{n_2 \times T_2} \qquad (1\text{-}2)$$

In this experiment, we will produce nitrogen gas by reacting a known amount of sulfamic acid (H_2NSO_3H), here as a limiting reagent, with an excess amount of sodium nitrite ($NaNO_2$) as shown in equation 1-3.

$$NO_2^-(aq) + H_2NSO_3H(s) \rightarrow HSO_4^-(aq) + H_2O(l) + N_2(g) \qquad (1\text{-}3)$$

By measuring the volume of water expelled by the nitrogen produced (ΔV), the number of moles of the nitrogen produced (n), the room temperature (T), and relevant pressures (P_{atm}, the atmospheric pressure; P_{H_2O}, the vapor pressure of water), we can determine the molar volume of nitrogen gas at STP (V_{STP}) by equation 1-4.

$$\frac{1\ (\text{atm}) \times V_{STP}\ (\text{L})}{1\ (\text{mol}) \times 273.15\ (\text{K})} = \frac{P_{N_2} \times (V + \Delta V)}{n \times T} = \frac{(P_{atm} - P_{H_2O}) \times \Delta V}{n \times T} \qquad (1\text{-}4)$$

Apparatus

Erlenmeyer flask (250 mL), Florence flask (500 mL), three-prong clamp (2), rubber tube and stopper, small glass test tube (7.5 cm × 1.2 cm, 2), beaker (400 mL), graduated cylinder (50 mL), plastic beaker (1 L), tweezers.

Shared: electronic balance, analytical balance, barometer, and thermometer.

Chemicals

Sulfamic acid, H_2NSO_3H

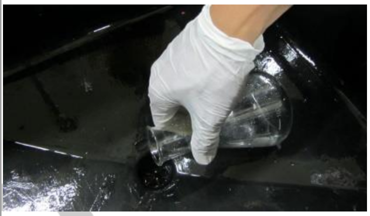
Sodium nitrite, $NaNO_2$

Procedure

Procedure		Illustration
1.	(1) Wash and oven-dry two small test tubes and allow them to cool to room temperature. (2) Measure and record the accurate weight of one of the tubes.	
2.	Carefully measure 1~1.1 g of H_2NSO_3H into the small test tube. Record its accurate weight.	
3.	(1) Weigh about 1~1.1 g of $NaNO_2$, record its accurate weight, and transfer it into the 250 mL Erlenmeyer flask. (2) Use a graduated cylinder to measure *ca.* 50 mL of DI water into the flask to dissolve it.	

4.	(1) Fill Florence flask B and 400 mL beaker D with water, up to the level as shown in Fig. 1-1(a). (2) Fill rubber tube C with water, clamp it with a pinch clamp, and then connect all apparatus. Carefully adjust the glass tube in flask B such that the end of the longer pipe nearly touches the bottom of the flask. Note: To fill rubber tube C with water, bring one end of the tube under the faucet until water flows out from the other end; then clamp it tightly with the pinch clamp.	 
5.	Loosen the pinch clamp to make sure that the rubber tube is filled with water and the water can flow freely from Florence flask B to beaker D while adjusting the height of beaker D. Then tighten the pinch clamp again.	
6.	With a pair of tweezers, carefully position the small test tube at the bottom of the tilted Erlenmeyer flask A. After the small test tube is positioned securely, stopper flask A tightly (Fig. 1-1(a)). Caution: The small test tube must not topple. Once it does, $NaNO_2$ will immediately react with H_2NSO_3H. Should this happen, wash the flask and tube with plenty of water and redo the procedure.	 
7.	(1) Loosen the pinch clamp and adjust the height of beaker D such that the water levels in beaker D and flask B are equal to balance the pressure of the reaction system in flasks A and B to the atmospheric pressure. (2) When the balance is achieved, tighten the clamp and discard the water in the beaker. (3) Measure the weight of the empty beaker. Note: Gently rotate rubber stoppers for *ca.* 15° to make sure they are tightly sealed.	 

8.	(1) Loosen the three-prong clamp on flask A to stand it upright on the table. The small test tube then topples, allowing the $NaNO_2$ solution in flask A to enter the tube and react with H_2NSO_3H in it. (2) Effervescence will be observed. Loosen the pinch clamp on rubber tube C at the same time, allowing water to flow out freely. Note: When the reaction just starts, do not swirl the Erlenmeyer flask vigorously to avoid violent gas generation, which may cause the rubber stopper to pop out.	 
9.	Swirl flask A gently until the reaction is complete and no more gas is produced. Note 1: Keep the outlet of the rubber tube underneath the water in beaker D all the time. Note 2: This experiment has a side reaction that produces reddish-brown toxic NO_2 gas.	
10.	Cool flask A in a water bath to room temperature.	
11.	Adjust the height of beaker D or flask B until the water levels in them are equal. At this time, the internal pressure of the reaction system is equal to the atmospheric pressure. Tighten the pinch clamp. Note: The calculation of experimental data is premised on keeping temperature and pressure constant before and after the reaction. Hence, the temperature must return to room temperature before the pressure is adjusted.	
12.	Measure the volume of water collected in beaker D by weighing method.	

13.	Measure and record the corresponding room temperature and atmospheric pressure.	
14.	(1) At the end of the experiment, pour off the reacted solution and rinse the glassware immediately to avoid the NO_2 evolved. (2) Wash the apparatus, clean up your benchtop, and complete the experiment.	

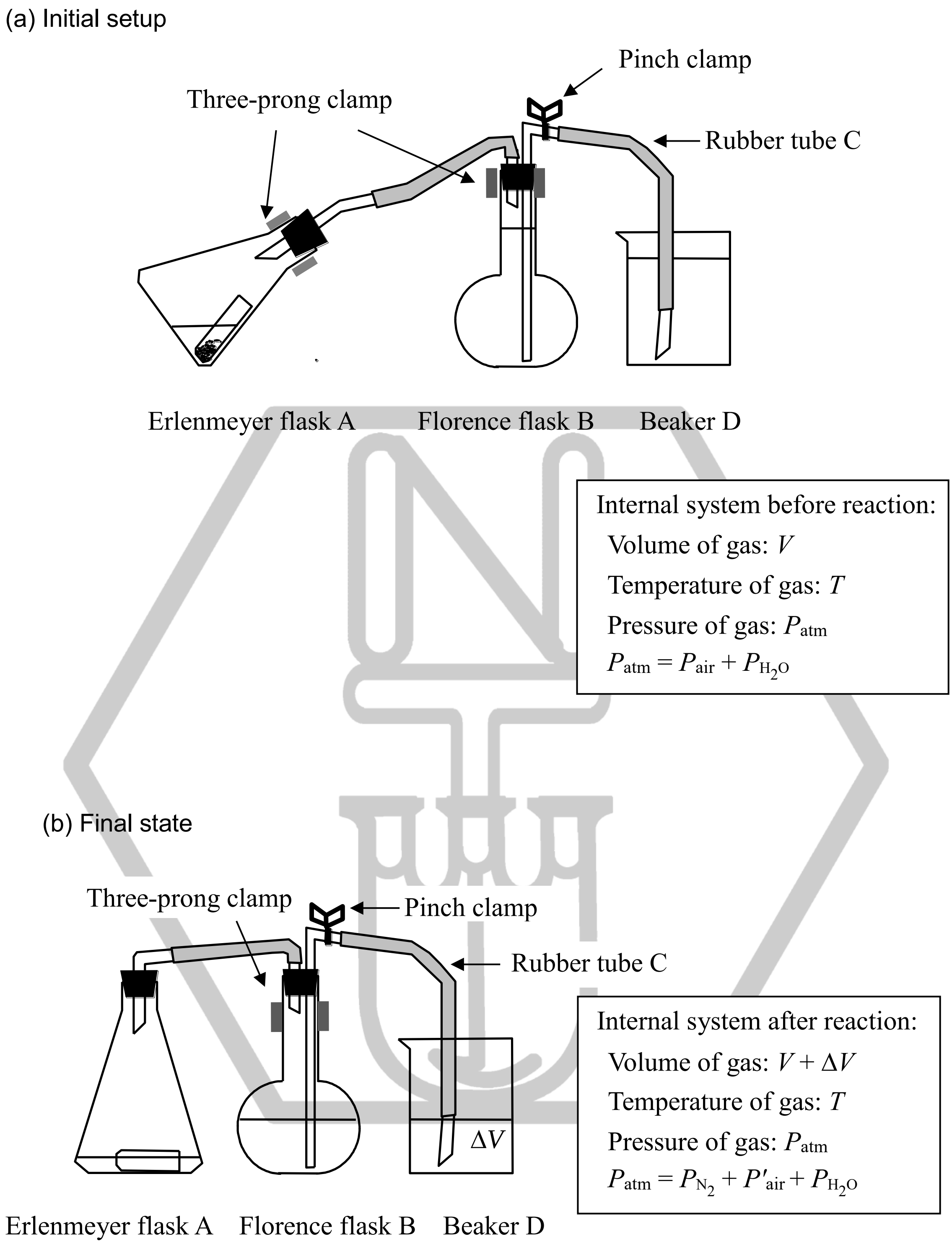


Figure 1-1 The apparatus for determining the molar volume of nitrogen