



# General Chemistry Laboratory

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## The Molar Volume of Nitrogen Gas



# Preparation

- Put on your lab coat and safety goggles
- Turn off your mobile phone
- Place your backpack in the drawer or the cabinet
- Put your prelab on the lab bench (hold it down with something heavy) for ATA to sign

## **Collect the following items**

- Two small test tubes (to be distributed by TA)
- An iron stand, two three-prong clamps
- 250 mL Erlenmeyer flask, 500 mL Florence flask, a rubber stopper with rubber tube and glass tube

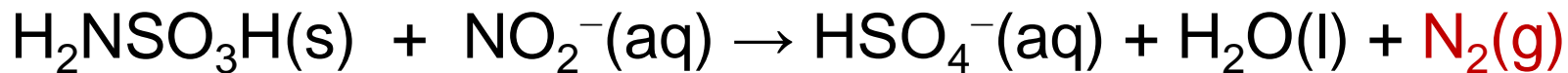


# Objective and Principles

- **Objective:** Using the ideal gas law to deduce the molar volume of nitrogen gas at STP
- **Lab techniques:**
  - Using an electronic balance to weigh chemicals
  - Measuring volume, pressure, and temperature
- **Definitions:**
  - Molar volume: the volume of a mole of substance
  - STP (standard temperature and pressure): 0 °C, 1 atm
  - For ideal gas, molar volume = 22.414 L at STP



# Nitrogen-Producing Reaction



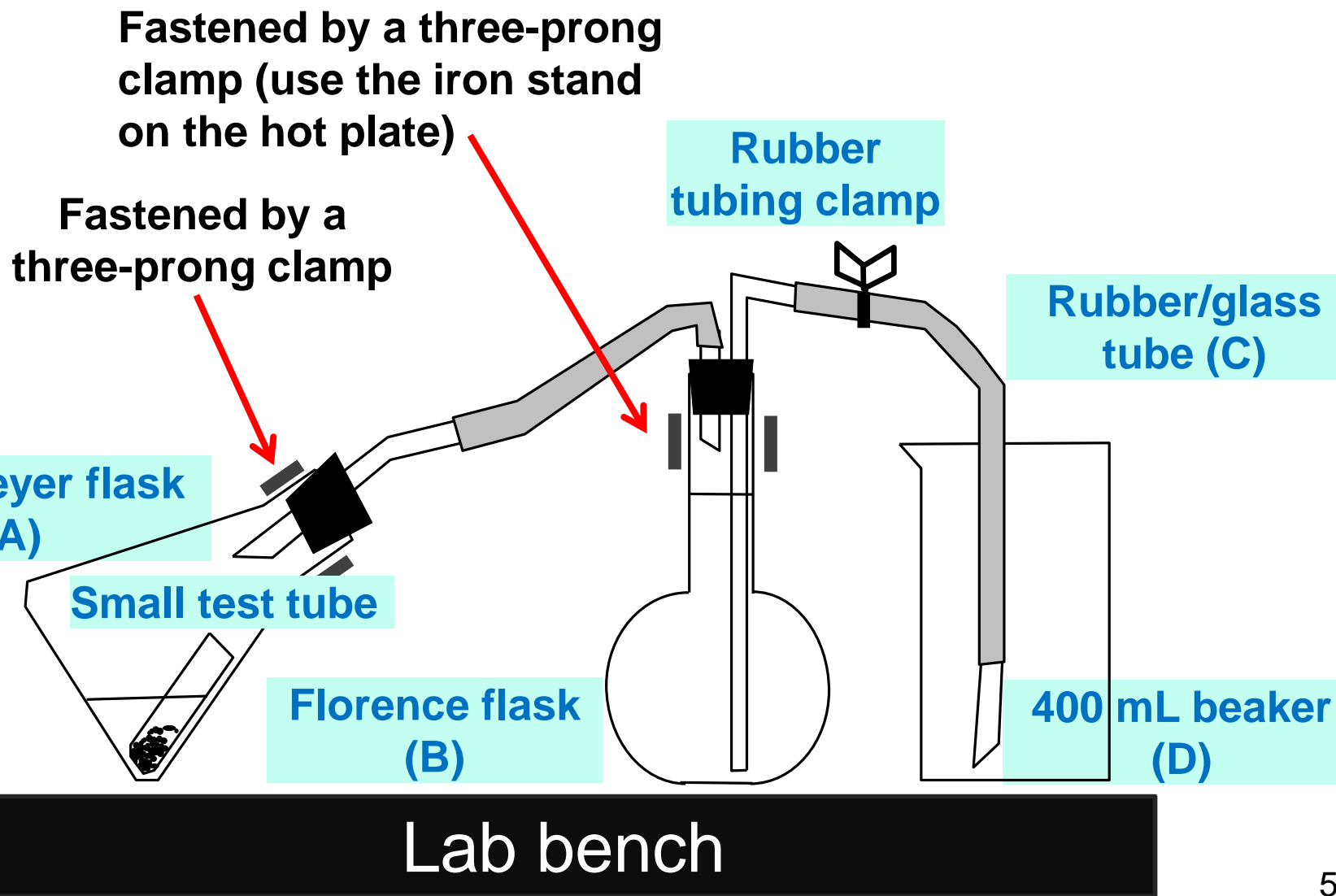
Initial:	$n_1$	$n_2$			
Change:	$-n_1$	$-n_1$	$+n_1$	$+n_1$	$+n_1$
Final:	0	$n_2 - n_1$	$n_1$		$n_1$

$(n_2 > n_1)$

- Sulfamic acid ( $\text{H}_2\text{NSO}_3\text{H}$ ,  $n_1$  mole) being the limiting reagent
- Sodium nitrite ( $\text{NaNO}_2$ ) being the excess reagent

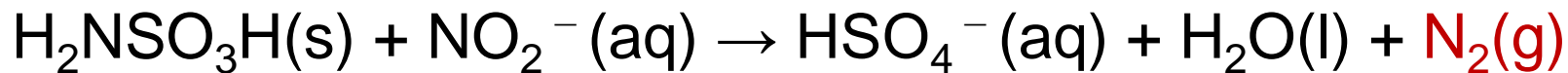


# Experimental Setup





# Nitrogen-Producing Reaction



- Sulfamic acid ( $\text{H}_2\text{NSO}_3\text{H}$ ,  $n_1$  mole) being the limiting reagent
- Sodium nitrite ( $\text{NaNO}_2$ ) being the excess reagent
- The molar volume of  $\text{N}_2$  at  $0^\circ\text{C}$  and  $1\text{ atm}$ ,  $V_{\text{STP}}$ , can be related to the volume of  $\text{N}_2$  ( $V_1$ ) at room temperature ( $T_1$ ) given the number of mole ( $n_1$ ):

$$\frac{1(\text{atm}) \times V_{\text{STP}}(\text{L})}{1(\text{mol}) \times 273.15(\text{K})} = \frac{P_{\text{N}_2} \times V_1}{n_1 \times T_1} = \frac{(P_{\text{atm}} - P_{\text{H}_2\text{O}}) \times \Delta V}{n_1 \times T_1}$$

*Mercury barometer*
*Vapor pressure of water*
*Volume of collected water*

*# mole  $\text{H}_2\text{NSO}_3\text{H}$* 
*Thermometer*



# Step 1/6: Measuring Chemicals

- Measure roughly 1 g  $\text{NaNO}_2$  and dissolve them with 50 mL DI water in the Erlenmeyer flask A
- Measure the weight of an empty small test tube ( $W_1$ ) using an analytical balance
- Place roughly 1.0-1.1 g  $\text{H}_2\text{NSO}_3\text{H}$  into the small test tube, record the accurate weight ( $W_2$ )



- ✓ Use a small beaker to hold the test tube
- ✓ Only the weight difference ( $W_2 - W_1$ ) matters



# Step 1/6: Analytical Balance



Place a small beaker  
(container) on the  
weighing pan



Close  
windshield  
& zeroing  
(TARE)



Display shows 0.0000 g  
(Deduction of container's mass)



Place test  
tube in &  
close  
windshield



$W_1: 7.5757 \text{ g}$   
(Mass of small test tube)

Take out the  
small test tube



Use the skinny end of a spatula to  
put ~1 g of  $\text{H}_2\text{NSO}_3\text{H}$  (ca. 4 times) 8  
into the small test tube



Place the test tube  
back in and close  
the windshield



$W_2: 8.6202 \text{ g}$   
(Mass of test tube and  $\text{H}_2\text{NSO}_3\text{H}$ )

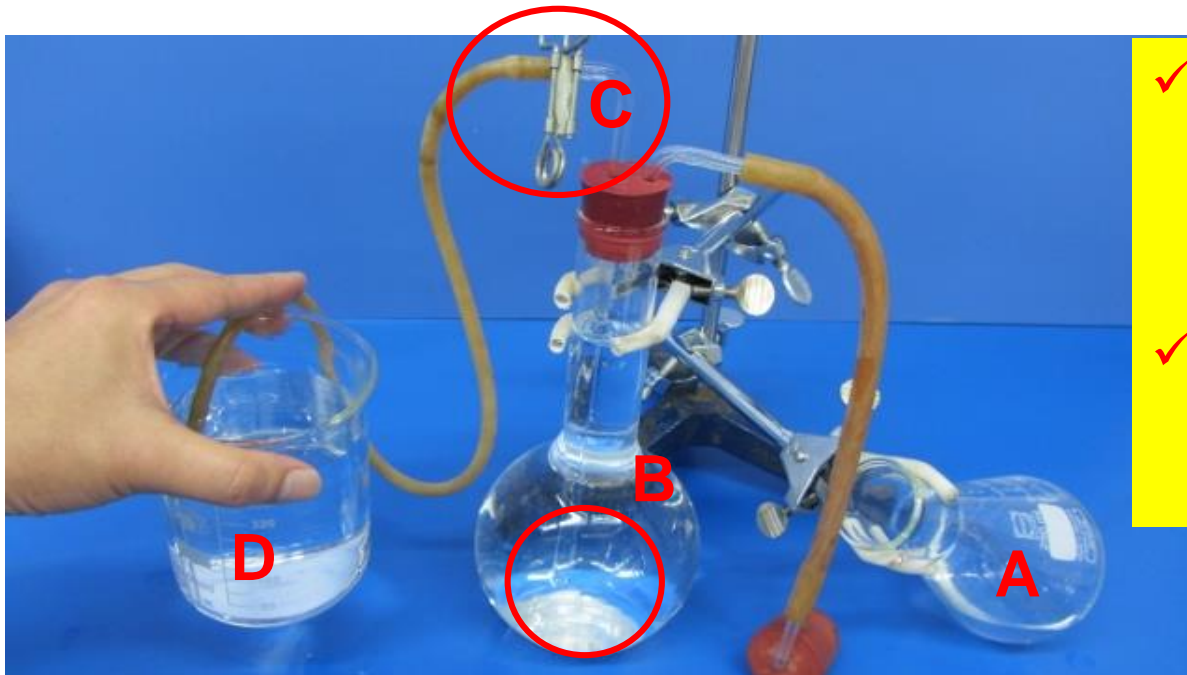
Mass of  $\text{H}_2\text{NSO}_3\text{H}$   
 $W_2 - W_1 = 1.0445 \text{ g}$   
(Five s.f.)





## Step 2/6: Check the Water Flow

- Fill both the Florence flask B and the beaker D with water
- Fill the rubber/glass tube C with water, apply the tubing clamp
- Use the rubber/glass tube C to connect B and D
- Loosen the tubing clamp and check if the water can flow freely between the two containers; re-apply the tubing clamp



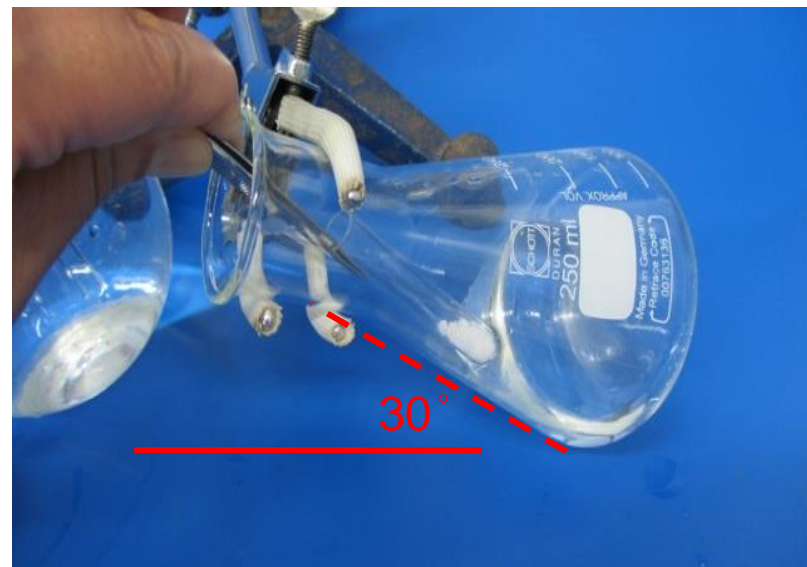
- ✓ The end of glass tube shaft should nearly touch the bottom of the Florence flask
- ✓ Check whether there are air bubbles in the rubber/glass tube



# Step 3/6: Set up the Reaction Vessel

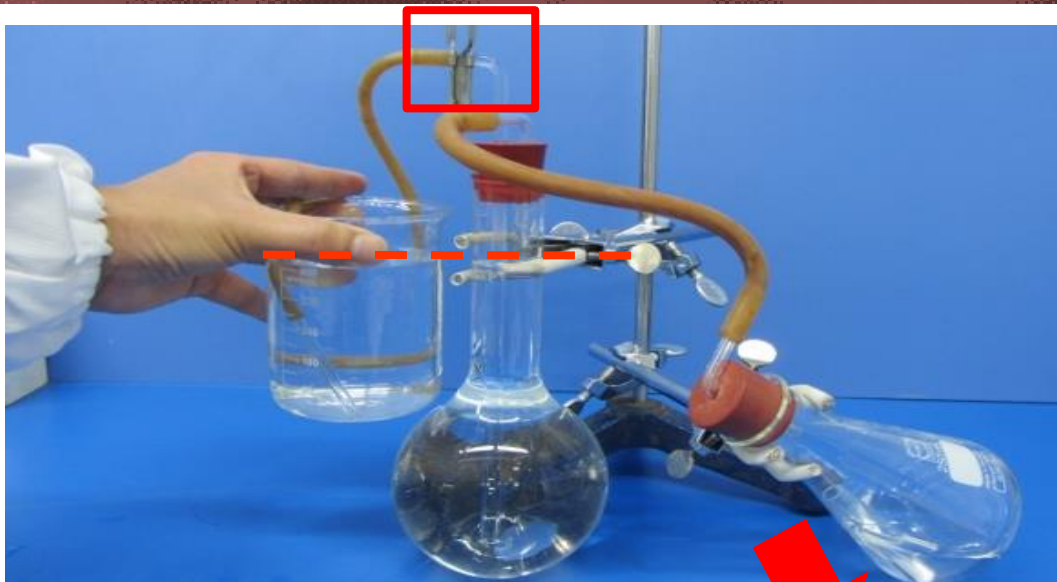
- Use a three-prong clamp to fix the Erlenmeyer flask A at a tilted angle
- Use a tweezer to place the test tube containing  $\text{H}_2\text{NSO}_3\text{H}$  at the bottom of Erlenmeyer flask A
- Install the rubber stopper onto the Erlenmeyer flask

- ✓ The small test tube must not topple
- ✓ Once it does,  $\text{NaNO}_2$  will immediately react with  $\text{H}_2\text{NSO}_3\text{H}$
- ✓ Should this happen, wash the flask and tube with plenty of water and redo the procedure.



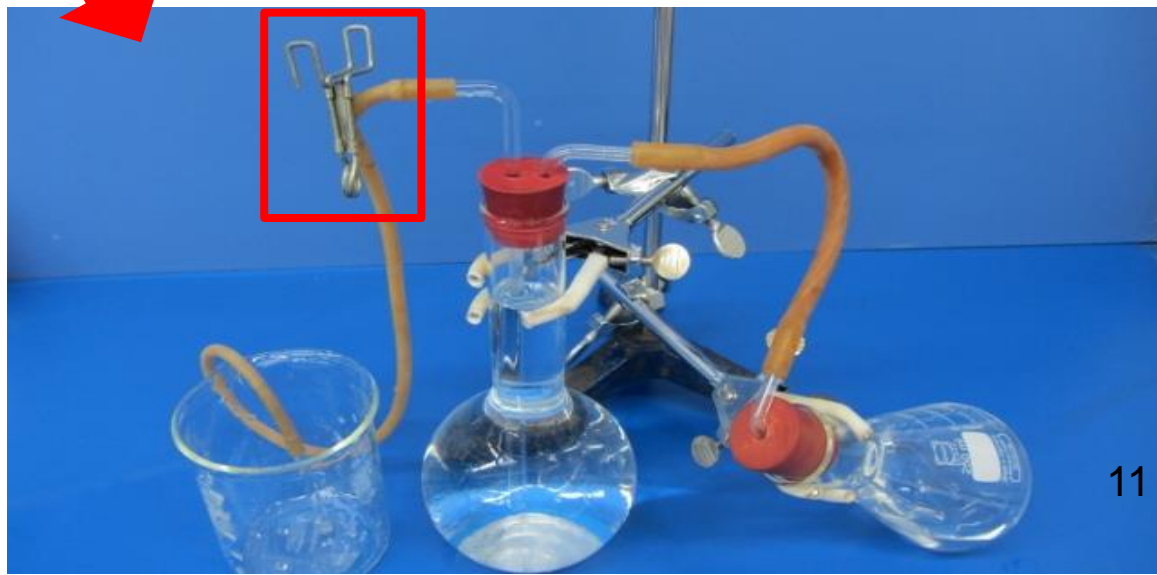


# Step 4/6: Balance the Pressure



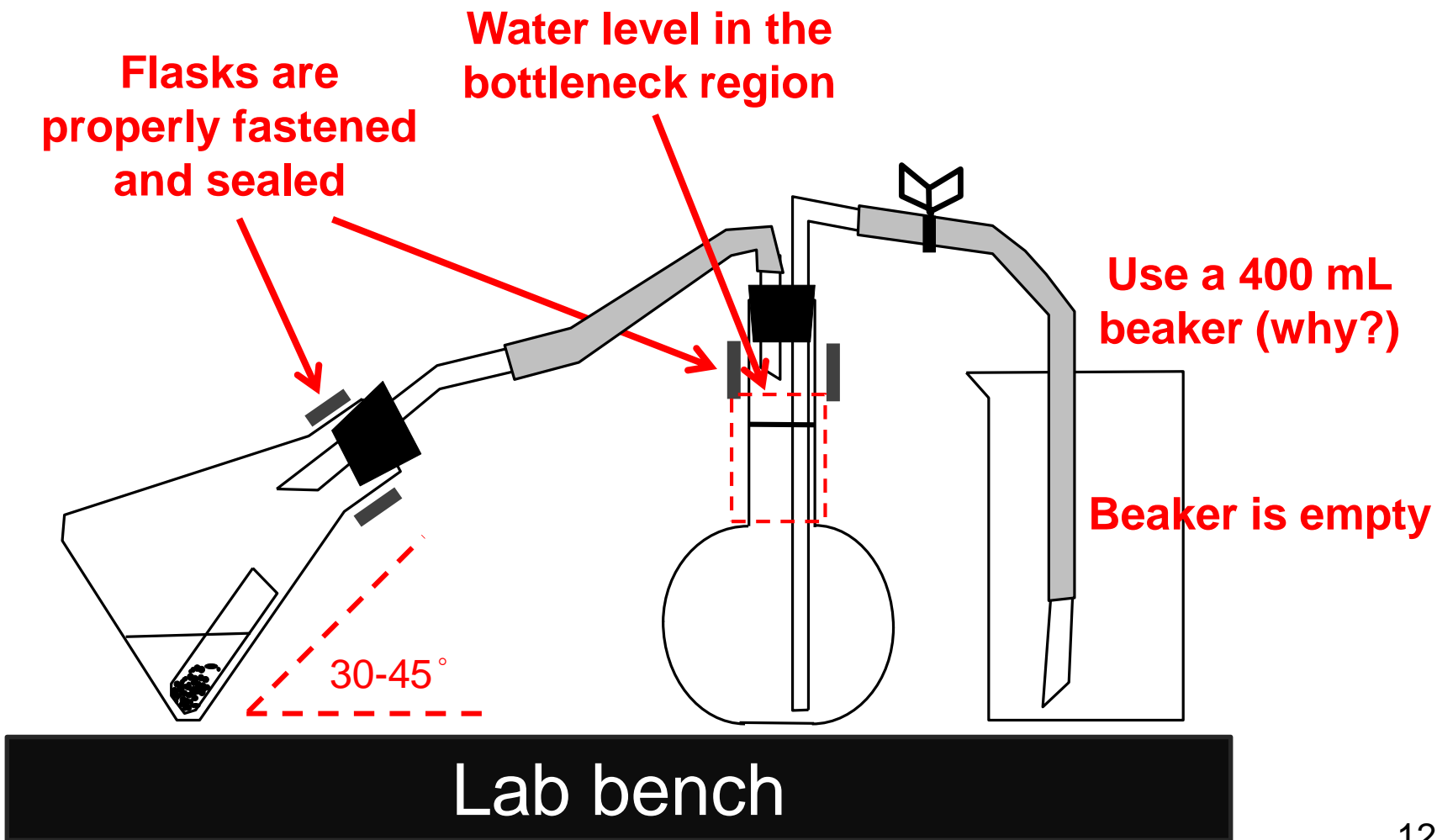
- Ensure all rubber stoppers are tightly fitted
- Loosen the tubing clamp and adjust the height of beaker D so that the water levels in flask & beaker are of the same height

- Tighten the tubing clamp and dispose all the remaining water in the beaker
- Measure the weight of the empty beaker





# TA/ATA Check Your Setup

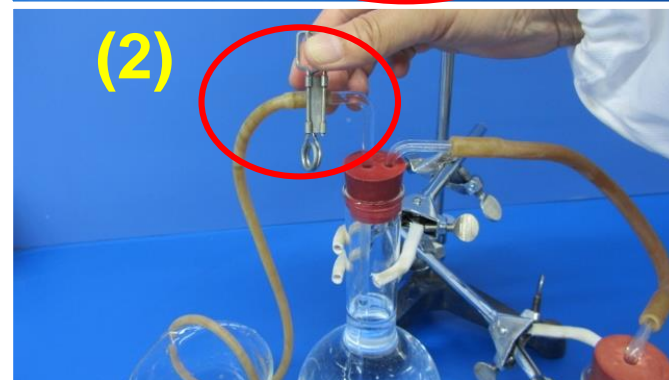




# Step 5/6: Start Generating Nitrogen

- 1) Loosen the three-prong clamp on Erlenmeyer flask, straighten the flask, and let the chemicals mix
- 2) Once bubbles appear, loosen the tubing clamp quickly
- 3) Swirl the flask gently and observe the water flow until the reaction is complete and no more gas is produced

- ✓ Do not twist the rubber tube, and keep outlet of the rubber tube underneath the water in beaker all the time
- ✓ Brown  $\text{NO}_2$  gas may be produced via a side reaction



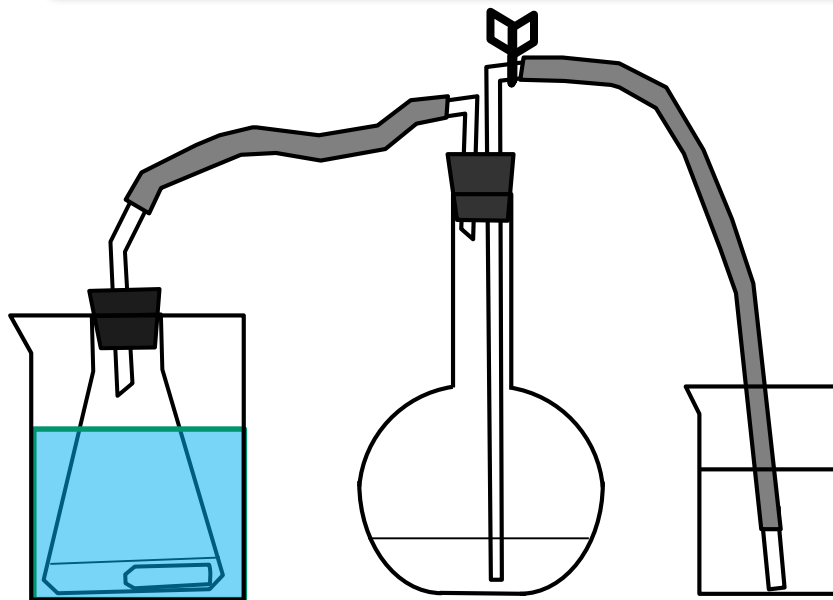




## Step 6/6: Adjust Pressure and Temp

- Place the Erlenmeyer flask in a tap water bath to cool to room temp
- Adjust the height of the Florence flask so that the water levels in flask & beaker are at same height
- Tighten the tubing clamp
- Measure the weight of the beaker and water collected
- Record the room temp and pressure,  $P_{\text{atm}}$
- Find vapor pressures of water,  $P_{\text{H}_2\text{O}}$  from appendix

Keep the pressure and temperature of reacting system same as room temp and ambient pressure before and after reaction



1 L Beaker as  
water bath

$\Delta V > 250 \text{ mL}$



# Lab Note and Data Analysis

- Use ball pens and avoid correction tapes
- Record operations and raw data (value + units)
- Changes that can be observed:
  - The appearances of reactants and products
  - What happens as reactants mixed together?
  - The reaction rate, exo- or endothermic reaction, color change.....
- Data analysis

$$\frac{1(\text{atm}) \times V_{STP}(\text{L})}{1(\text{mol}) \times 273.15(\text{K})} = \frac{(P_{atm} - P_{H_2O}) \times \Delta V}{n_1 \times T_1}$$

$$V_{STP} = ?$$

- ✓ Use the correct significant figures and units to show the answer
- ✓  $T_1$  in K
- ✓ P in atm



# Clean-Up and Check-Out

- Brush clean and return the small test tubes
- Pour the solution waste directly into the drain
- Tuck the lab stools underneath the lab bench
- Clean up the lab bench and check personal equipment inventory (have an associate TA signed the check list)
  
- This is a **Brief Report** experiment:
  - Complete calculation using correct significant figures
  - Give a conclusion of your experiment
  - **Hand in prelab/lab note/report together to the TA**
  
- Groups on duty shall stay and help clean up the lab





# Final Report (Brief Version)

- List the detailed calculations in the blank space (pay attention to unit conversion!)
- Conclusion: Use 1-2 sentences to summarize your experiment formally
- Need not answer the discussion questions in the report sheet (Q1 is the bonus question for the prelab)

## Experiment 1

### MOLAR VOLUME OF NITROGEN GAS

#### I. Experimental Data and Results (show all calculations)

1. Weight of small test tube ( $W_1$ ) \_\_\_\_\_
2. Weight of small test tube with  $H_2NSO_3H$  ( $W_2$ ) \_\_\_\_\_
3. Weight of  $H_2NSO_3H$  ( $W_2 - W_1$ ) \_\_\_\_\_
4. Weight of  $NaNO_2$  \_\_\_\_\_
5. Weight of empty beaker ( $W_3$ ) \_\_\_\_\_
6. Weight of beaker and water collected ( $W_4$ ) \_\_\_\_\_
7. Volume of water expelled ( $\Delta V$ ) \_\_\_\_\_
8. Atmospheric pressure ( $P_{atm}$ ) \_\_\_\_\_
9. Room temperature ( $T$ ) \_\_\_\_\_
10. Vapor pressure of water at this temperature ( $P_{H_2O}$ ) \_\_\_\_\_
11. Number of moles of  $NaNO_2$  \_\_\_\_\_
12. Number of moles of  $H_2NSO_3H$  ( $n$ ) \_\_\_\_\_
13. Molar volume of nitrogen gas at STP (show your calculations):

(Refer to question 1)

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



# T3.1 – Mercury Barometer

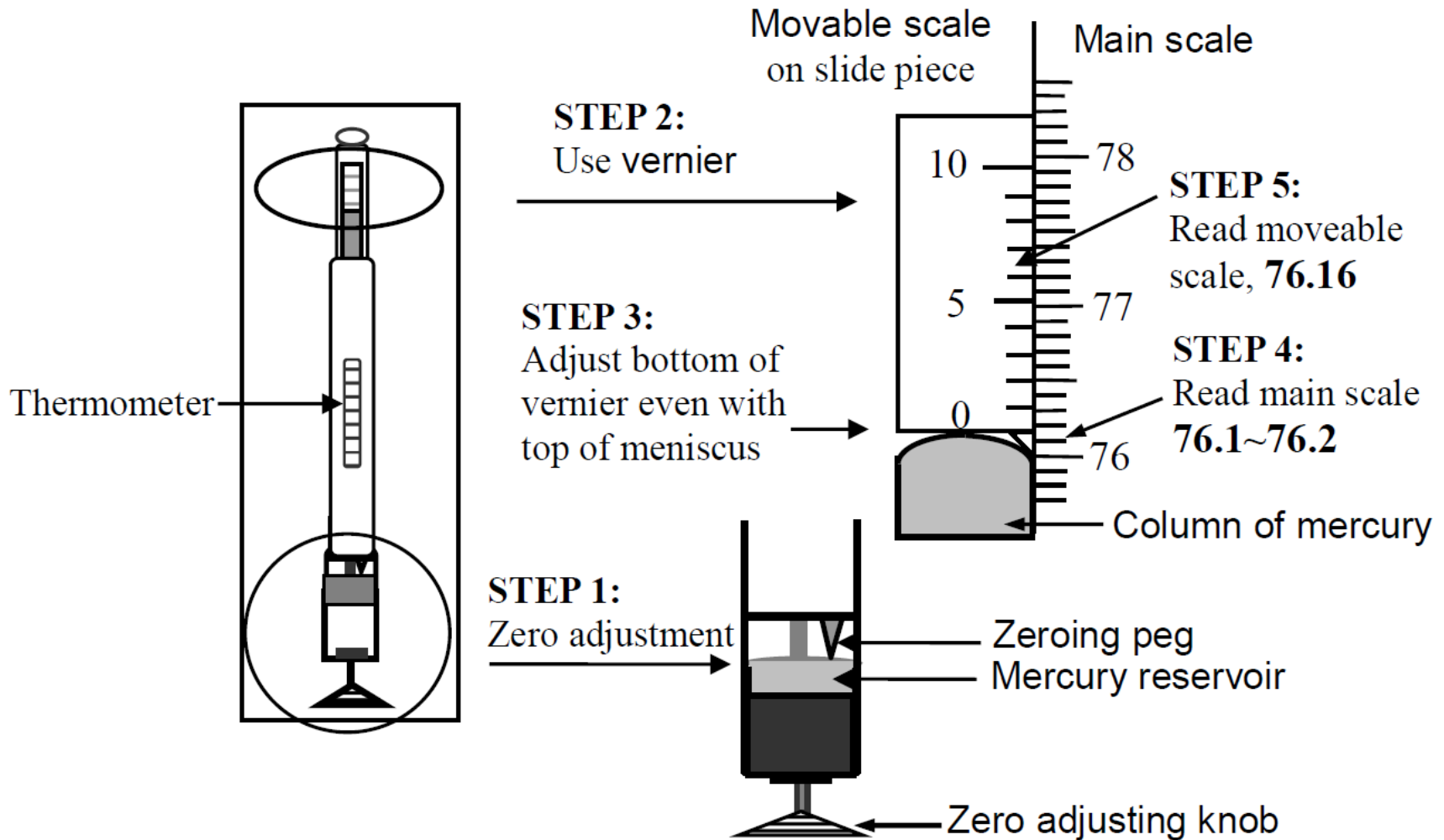


Figure T3-1 Illustration of mercury barometer



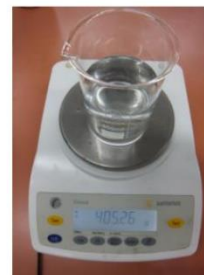
# T3.2 – Mercury Barometer

- Zero adjustment: Rotate the **Zero Adjustment Knob** at the bottom of barometer to align the tip of **Zeroing Peg** with the top of mercury reservoir
- Read the mercury column height with a **Vernier Scale**:
  1. Adjust the **Slide Piece** so that its bottom is even with the top of mercury meniscus
  2. Read the main scale on the right: In Figure T3-1, the bottom of the slide piece indicates the height of mercury column is between 76.1 and 76.2 cm
  3. Read the movable scale on the left: The percentile of measurement is given by the line that aligns with the mark on the main scale. This value is 6 in Figure T3-1, therefore the measured atmospheric pressure is 76.16 cm-Hg
- For more precise measurement, one should make temperature correction (refer to the manual of barometer)



# T9 – Electronic Balance

- Unless instructed, do not move the balance so that proper calibration is maintained
- Do not overload the balance (the maximum load is 610 grams for *electronic balance*, and 210 grams for *analytical balance*)
- Before use, warm up the balance for at least 30 min and ensure that it is level and clean
- Do not put chemicals directly on the weighing pan – use a folded weighing paper, a weighing boat, or a beaker (mind the weight limit) as container
- Close all windshields on the *analytical balance* before zeroing and recording values
- Maintain the tidiness inside and outside the balance; use the provided soft brush to clean spillages
- Do not weigh hot objects as the convective airflow will affect the measured mass



**Electronic Balance** (resolution 0.01 g)



**Analytical Balance** (resolution 0.0001 g)



# T10 – Weighing Chemicals

- Read the label on the chemical bottle carefully before proceeding to weigh
- For solid chemicals, place a folded weighing paper or a beaker on the electronic balance to hold chemicals. Use a clean and dry spatula to move chemicals
- For liquid chemicals, use a clean and rinsed dropper pipet
- Unless specifically instructed, never return any excess chemical to the original bottle to avoid contamination – use the designated waste bin
- Maintain the tidiness inside and outside the balance – move appropriate amount with spatula to avoid any spillage, and use the provided soft brush to clean scattered chemicals
- Close the cap of chemical bottle immediately after use

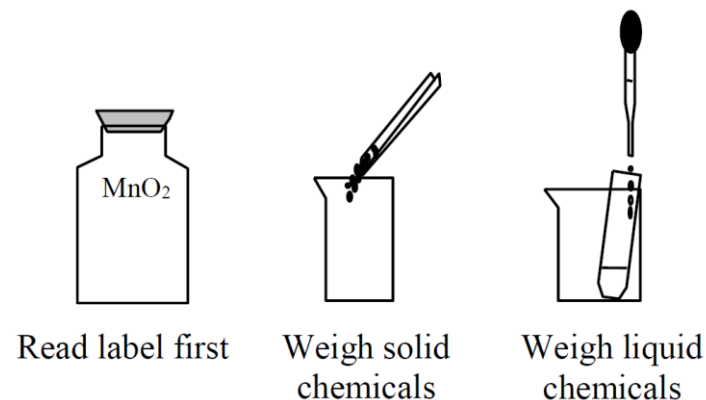


Figure T10-1 Weighing chemicals